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—OF THE—

American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries.

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JOURNAL
OF THE
AMERICAN CERAMIC SOCIETY

A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 1 **July, 1918** **No. 7**

EDITORIALS.

LOOKING FORWARD.

With the practical ending of the war we look back with pride at the achievements of the Society and its members in the solving of the problems arising in its successful prosecution and in meeting the difficulties arising in the normal conduct of the various branches of the industry. The ceramic industry has kept pace with the advancement of the other great industries during this period of trial and we have seen the successful placing of several new and important products on a firm and lasting manufacturing basis in this country—notably the production of optical glass, chemical glassware, chemical porcelain, etc.

Looking forward, we see some new and some old problems which remain to be faced.

The scarcity and steadily rising cost of fuel of all kinds is stimulating interest on the part of our manufacturers in improved types of kilns and methods of combustion. The suggestion that at our annual meeting in February we have a session at which the merits of the various types of patented dryers, gas-producers, tunnel kilns and continuous kilns be discussed from a technical and economic standpoint, is an excellent one and it is to be hoped that an opportunity for a discussion of this kind will be afforded.

In this number of the JOURNAL, Dr. Ries gives an excellent résumé of the available and prospective sources of high-grade clays in the United States. Linked as it is with the question of the production of high-grade pottery wares from all American

materials, the problem of the location of new deposits of American kaolins and of improvement in the methods of their purification should not cease to receive the attention of our geologists and technologists. We are faced with numerous other problems awaiting attention and solution.

PROFESSIONAL DIVISIONS.

As a culmination of the discussion which has taken place of late in reference to the formation of industrial divisions of the American Ceramic Society, the Board of Trustees has recently affirmed the following motion:

Professional divisions may be formed in the following ways:

1. "When the initiative in the formation of divisions is taken by members who are interested, a petition may be presented by not less than ten members in good standing, of whom three or more shall be Active, who are interested in some phase of ceramic work sufficiently broad to warrant the formation of a special division. This petition shall go to the President who shall then appoint a representative committee to consider the advisability of forming such a division, and to proceed with the organization if the decision is favorable.

2. When the initiative is taken by the Board of Trustees in order to stimulate the growth of the Society, the President shall appoint a representative man to furnish the initiative. He shall select his own committee."

The steady growth and enlargement of the scope of the activities of the Society warrant this action by the Board at this time. Our Society has outgrown the old idea of each member having a broad interest in all kinds of ceramic work. If we are going to maintain solidarity in technical ceramics, as it has been in the past through the American Ceramic Society, we must furnish opportunity for specialization. Professional divisions will furnish this opportunity.

It is to be hoped that there will be no delay in the organization of the respective professional divisions. The problem of the formation of several divisions of the Society at once is not a difficult one and is only a question of which branches of the industry offer the most fertile fields for stimulation and growth. It would appear that the early organization of the glass and pottery divisions is particularly desirable.

In the past the importance of quickening the interest of a larger proportion of the glass industry in the Society has been too little recognized. The technical achievements and awakened interest in this highly important branch of the ceramic industry—through the demands of the war for special glasses—should not be permitted to die out.

It is needless to mention the importance of the active support of the pottery industry to the future welfare and advancement of the Society and the necessity of stimulating research on the problems arising in this basic industry. This is especially vital in view of the ending of the War and the consequent looming up of the old and serious problem of again meeting the competition of the foreign wares.

The organization of professional divisions for the other branches of the industry is of equal importance and in passing we may hastily mention the advisability of the formation of divisions on enamels, refractories, abrasives, etc.

The securing of an adequate supply of professional papers for the issuance of a Journal of even the present modest size has proven somewhat difficult. Through the coöperation of the Professional Divisions and Local Sections there should be an adequate flow of varied technical contributions which will assure the future success of the Society's publications.

ORIGINAL PAPERS AND DISCUSSIONS.

THE OCCURRENCE OF HIGH-GRADE AMERICAN CLAYS, AND THE POSSIBILITIES OF THEIR FURTHER DEVELOPMENT.[1]

By H. Ries.

Introduction.

Although the United States has always ranked foremost among the countries of the world as regards its mineral resources, nevertheless, a not inconsiderable quantity of certain raw materials has been obtained in the past from foreign sources. This was no doubt due in part to the comparative ease and cheapness with which some of these could be obtained, and also to the fact that many of them were of high grade, the result being that domestic consumers in many instances imported certain necessary mineral products rather than search for them in this country.

With the beginning of the great war, certain mineral products, hitherto obtained from central Europe, were almost immediately shut off, while later, owing to the scarcity of ships, it became necessary to further curtail the imports—even of those that could still be imported from neutral or allied countries.

This curtailment of imports has in some respects been beneficial to the United States, for it has stimulated the search for, and development of, good deposits of useful minerals within our boundaries, and it has, moreover, shown the possibility of using low-grade domestic materials hitherto disregarded.

To quote one case outside of the field of clay. For many years we have imported emery from Turkey and Greece. Since

[1] By permission of the Director, U. S. Geological Survey.

the war we have begun the development of a high-grade emery field in Virginia, which apparently contains a large tonnage of this abrasive.

While the clay imports form but a small percentage of the total imports of mineral products, nevertheless, taken by themselves they make up no small tonnage, this in 1916 amounting to 298,866 short tons, valued at $1,504,233, and in 1917 to 239,318 short tons, valued at $1,442,059.

The clays which have been imported into the United States include those from central Europe which were used in the manufacture of glass pots, graphite crucibles, enamels, etc., and the china clays and ball clays of England, much sought after by manufacturers of whiteware, electrical porcelain, paper, etc.

While it is true that the supply of the English clays has not been completely shut off, it has recently been diminished on account of dock repairs at Fowey, and still further restricted at a very recent date (August), by an order of the British government—essentially prohibiting the exportation of clay in British bottoms to Boston, New York and Philadelphia.

With the shutting off, therefore, of so much material of value to the ceramic industry, it becomes imperative to determine what means can be, or have been taken in this country to replace imported clays, or what the possibilities are for increasing the output of those high-grade ones now being obtained.

It is, of course, well known that kaolin, ball clays, paper clays, and glass-pot clays have been mined in this country for some time, but nevertheless, they have not fully met the domestic demand in two ways: firstly, there has been an insufficient quantity and secondly, many manufacturers have claimed that, for certain purposes, it was desirable, if not necessary, to include some of the foreign clays in their mixtures—or in special cases to use them exclusively. This, therefore, brings up two problems, *viz.:* first, can the supply of those clays already known and being mined be increased, and second, can we find additional supplies of the types already known in this country, or deposits of new types?

To accomplish this requires two distinct lines of investigation, geologic and technologic.

The geologic investigation serves to show the areas in which new supplies may be looked for, their probable extent, size and, therefore, tonnage. Thus, it saves the prospector and miner much time and money in his search. It also serves to give information regarding the character of the raw materials, their visible impurities, etc.

The technologic study takes up the further development of the problem and attempts to work the newly discovered materials into different mixtures, or possibly find new uses for those deposits of clay already exploited.

It is to the first of these that I shall give especial attention—touching the latter but briefly.

High-Grade Clay Deposits of the United States.

Any one familiar with our sources of raw materials used in the manufacture of the high-grade products mentioned above, knows that most, if not practically all, of them are located in the eastern half of the United States, and while some of value may occur in the far west, they have not been drawn upon by the factories of the eastern and central states—due probably because of the long hauls and consequently the high freight rates.

During the past summer and spring, the writer has had occasion to visit most of the localities in the eastern half of the United States which are capable of supplying raw materials of high grade to the industries already referred to, and in the present paper it is proposed to outline briefly what the chances are for obtaining supplies and also to touch briefly on their present uses.[1]

The area under discussion embraces that portion of the United States lying east of the Mississippi and in addition portions of Arkansas and Missouri.

For purposes of convenience of discussion, we may divide this region into several geographic subdivisions as follows:

1. The Atlantic Coastal Plain belt, extending from New Jersey to Florida and Mississippi inclusive.

2. The so-called Embayment Area, reaching from northern Mississippi and Texas northward into southern Illinois and in-

[1] The detailed results of this work will appear in a bulletin of the U. S. Geological Survey.

cluding western Tennessee and Kentucky, southern Illinois, southeastern Missouri, and eastern Arkansas.

3. The Piedmont plateau and mountain belt which lies just west of the Atlantic Coastal Plain belt and extends from Connecticut to northwestern Alabama, and westward to the Appalachian mountains.

4. The Coal Measures region of the central and southern states.

5. The Missouri flint-clay areas.

1. **The Atlantic Coastal Plain Belt.**—This region is underlain by a series of more or less unconsolidated sands and clays, which dip gently seaward—the most valuable clays having been found almost exclusively in the Cretaceous formation, or oldest one of the series, the only exceptions being certain localities in Georgia and Florida. This means, therefore, that the high-grade clays so necessary to many industries during the war are to be sought for mainly along or towards the inner side of this belt.

In tracing this formation from New York southward to Georgia and westward to Mississippi, we find that it shows considerable variation both as to the character of material and the possible tonnage.

The areas of clay in New York are small and of value chiefly for fire brick, but the extension of this clay belt across New Jersey is well known; indeed, the products of the Woodbridge and neighboring clay pits are so familiar to us as to need no further mention. Continuing to the southward, the same belt crosses Maryland, but shows an entirely different character and is of diminished importance. It is true that along some of the rivers flowing into Chesapeake Bay, such as the Severn, white refractory clays have been reported and do exist, but the deposits are lens shaped and, so far as discovered, are not of large tonnage. There are also, in this same belt, some buff-burning clays of moderate refractoriness which have varied uses in the metallurgical industries, but the individual masses are small and indefinite. The clay from some of these localities is considered adapted for use in the crucible trade.

In Virginia, the Cretaceous for mation carries, so far as accessible, no high-grade clays, and the same is true in North Carolina,

but then to the southward, in South Carolina and Georgia, this same formation develops a series of clay beds of striking character and high commercial value. We refer to the deposits of white clays—variously known as kaolins and plastic kaolins. The materials at the present time are widely used in the paper trade and to a lesser extent in the manufacture of whiteware, electrical porcelain and wall tile.

These plastic kaolins are found in certain areas in Bibb, Twiggs, Wilkinson, Washington, Glasscock, Jefferson and Richmond

FIG. 1.—Removing sandy overburden from white clay with steam shovel at Gordon, Georgia.

Counties of Georgia; and in Aiken, Edgefield, Lexington, and Kershaw Counties of South Carolina. In Georgia, the clay is worked chiefly in the Dry Branch and McIntyre districts, while in South Carolina, the developments have been chiefly in the Aiken district.

Those of Georgia are usually washed and yield a high percentage of washed product, while those of South Carolina are put on the market mostly in the un-washed but pulverized form.

In view of the widespread demand for these clays, a few words may be said regarding them.

These clays are always in the form of lenses, overlain usually by iron-stained sands. The lenses are of variable size, some of them being large. The overburden of iron-stained sand varies from 3 to 30 feet. The clays are worked at some 15 localities, but there are, no doubt, many other deposits not yet developed, so that the production could be expanded by this means. It

FIG. 2.—Excavating white clay with steam shovel at McIntyre, Georgia. The overburden is cross-bedded iron-stained sand.

could be still further increased by working the washing plants up to their capacity. This latter would mean a production probably 30 per cent greater than at present. But here we encounter the question of labor supply and transportation facilities—both of which must be improved to aid in further output.

Certain of the deposits in Georgia contain beds of bauxite or bauxitic clay associated with the white clay.

The Cretaceous formation swings around to the westward and is found in Alabama and northeastern Mississippi. There are white clay lenses here, possibly not as extensive as the Georgia and South Carolina ones, and undeveloped. Moreover, they occur in a higher geologic horizon than those of Georgia.

Those in Alabama are found chiefly near Marion, while those of Mississippi are distributed around Iuka, Tishomingo County. The clay lenses in these two states are probably not as large as the

FIG. 3.—White clay overlying bauxite in Sweetwater mine, near Andersonville, Ga. Railroad tracks at upper limit of bauxite. White clay overlain by iron-stained sands.

Georgia and South Carolina ones, nor are the materials always as plastic.

East of the Georgia belt, there are some white clays in the higher lying Tertiary formations, but they have not been utilized. One of the best known overlies the bauxite in the Sweetwater bauxite pit, $5^1/_2$ miles east of Andersonville, Sumter Co.

There is also the well-known white plastic clay from Florida,

which is worked in Putman and Lake Counties, at points about 60 miles apart, but it probably underlies a considerable area in between and there is a possibility of much greater production.

2. **The Embayment Area.**—The coastal-plain formations, already referred to, swing northward from northwestern Mississippi and form a belt extending northward up the Mississippi Valley into southern Illinois. The belt also extends for some miles to the east and west of the Mississippi River. It is so-called because

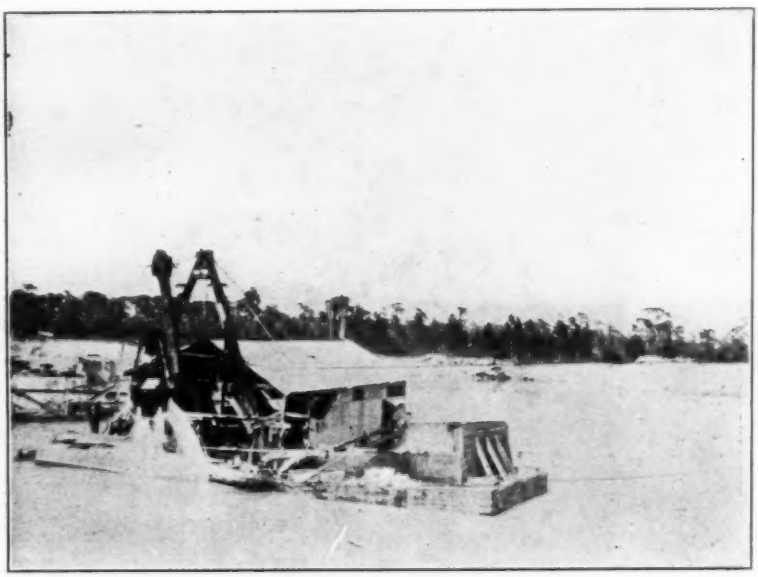

FIG. 4.—Dredging clay at Edgar, Florida.

the Cretaceous and Tertiary formations found here were deposited in a great northward reaching embayment of the sea, and it is here that some of our most important clays are found and where there is, we believe, great possibilities for future development.

Beginning at the south, we have in northern Mississippi several important clay formations which deserve careful investigation, but we cannot speak definitely regarding all of them as yet.

The first of these areas lies in the western half of northern Mississippi—the formation of Tertiary age, known as the Wilcox, containing a great series of white, grayish white, and light pink clays.

If we refer to a geologic section across this portion of the state, we notice that the beds dip westward. Consequently, those of the west half of the area will not be found in the eastern half because they do not exist there—having been eroded—and con-

FIG. 5.—Clay pit in Wilcox formation near Holly Springs, Miss. To the right of view the sand rises almost to the surface. At the left of view the clay rises to the surface. This shows the irregular occurrence of the clay in this region. Dark overburden is loam.

versely, those of the eastern half will be found only at considerable depths in the western half.

We separate this area into two halves because the clays are somewhat different. The western half yields clays of tough nature, high plasticity, buff-burning character, dense burning nature, and often high transverse strength. They vary in their

refractoriness. Some are dark colored—due to the presence of finely divided coaly material. The better ones are of the types that can be used in glass-pot mixtures, graphite crucibles, and in the bond of abrasive wheels. Others can be employed in stoneware, etc.

The clays of the eastern half occur in lenses, which sometimes pass rather rapidly into sand beds. The clays themselves frequently show thin layers of sand. They are now used only

FIG. 6.—Pit of refractory bond clay ten miles west of Enid, Miss. The overburden is loam.

for common stoneware, but their value after washing deserves to be considered. The silica content is high, and the iron oxide in published analyses ranges from 1.5 to 4.5 per cent. We question, however, whether we have here a grade of clay as good as those in the western half of the area.

The Tallahatchie county clays, although known for two or three years, are not yet fully developed and we believe that we have here an important clay region for the crucible and glass pot

trade, the demand for which is increasing steadily. The output can be greatly increased by better mining and by better roads and transportation facilities. It is curious that these clays should have remained undeveloped so long, but this may be due to the lack of outcrops, for the clays are usually covered by a sandy loam of varying thickness. However, their white outcrops in the bluffs bordering the Mississippi River bottoms should have attracted attention long ago.

FIG. 7.—Erosion gullies near Huntington, Tenn., showing natural exposure of the light-colored Lagrange clay.

Proceeding northward from Mississippi, through Tennessee and Kentucky, we have the well-known clay belt which has supplied us with ball clays and other grades. The main formation of this belt, known as the Lagrange division of the Tertiary, is really an extension of the Wilcox formation of northwestern Mississippi, but the change in the character of the contained clays is as great as that noted from New Jersey to Maryland. Only in the Southern part of Tennessee, as around Lagrange,

do we find clays approaching the northern Mississippi ones in character. Moreover, within the Lagrange formation, the best clays have been found near its eastern boundary—which extends from the southwestern part of Hardeman County, north-northeastward through Chester, Madison, Henderson, Carroll and Henry Counties. As we proceed westward, the covering of surface material becomes heavier and the clays will be found only under deeper cover.

FIG. 8.—Digging ball clay near McKenzie, Tenn. Shows the heavy overburden that is removed.

The Lagrange is not the only clay-bearing formation in Tennessee and Kentucky, there being three others forming narrow belts to the east of it, *viz.*, the Porters Creek, Ripley, and Eutaw, but these are, so far as known, of minor importance. No high grade clays have been developed thus far in the western part of the Lagrange area—although ball clay is reported from Eads, Shelby County.

The two questions that are of practical interest are, first, whether the production of these high-grade Tennessee and Kentucky clays can be increased, and second, whether any new uses have been found for them in recent years.

Firstly, as to production. This can be increased by improved labor and transportation facilities—a difficult matter to adjust at the present time. It can also be stimulated by the development of new deposits, which calls for careful prospecting. The

FIG. 9.—Pit of sagger clay near Henry, Tenn. Overlain by orange and white sand.

clays occur in lenses, many of which do not cover more than 10 or 12 acres, but some of which are considerably larger. These lenses are invariably covered by from three to forty feet of sandy overburden, and hence, the clay shows natural exposures only in gullies or stream banks. The fact that another clay lens is known to be near a developed one does not necessarily insure it being of the same quality, indeed, even in any one pit, there may be 7 or 8 different grades of clay.

The indications are that a considerable quantity of undeveloped clay may still remain in this region, but the larger proportion of better grades is probably to be found in the northern half of the Tennessee-Kentucky belt.

With regard to the second point—that of uses—these have expanded considerably since the area was first developed and we may say that they are now used in the manufacture of white-ware bodies, electrical porcelain, glass pots, crucibles, abrasive wheels, enamels, saggers, etc.

FIG. 10.—Pit of ball and sagger clay near India, Tenn. This pit supplies no less than three grades of clay and the overburden is gravelly material.

The same general formation that carries the ball-clays of Kentucky and Tennessee is found west of the Mississippi in Arkansas, but practically no development has gone on there and it affords a good field for the clay miner to prospect in. One exception should be noted, this is near Lester, Ark., where a bed of clay averaging 5 to 6 feet in thickness is found underlying a canneloid lignite. Tests which have been made of this material

show it to be adapted for use in glass-pot manufacture. There is an excellent chance to develop more clays in this area by further prospecting. Indeed, the formation carrying this clay extends northward in Arkansas and southward into Texas—where the beds of lignite are often underlain by beds of plastic refractory clay.

In the embayment region, there still remains to be mentioned the clays from southern Illinois which lie at the northern end

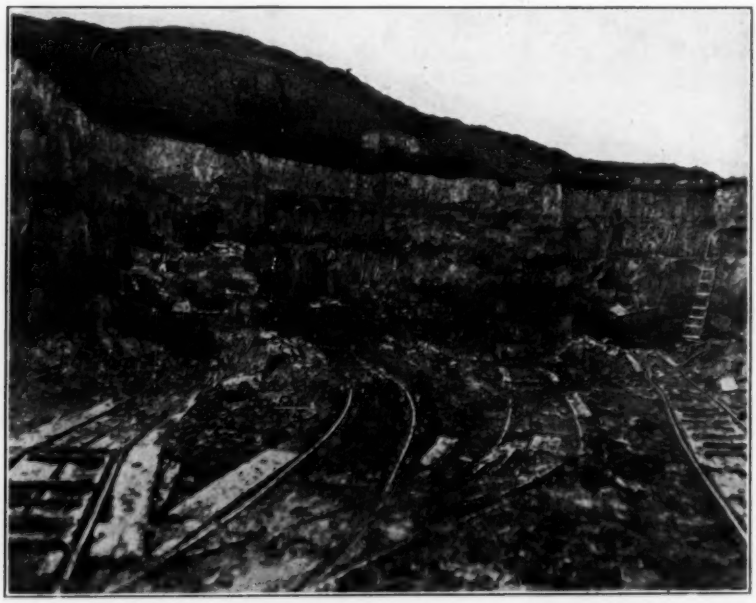

FIG. 11.—Clay pit at Pryorsburg, Ky., showing no less than four grades of clay in the face of bank. Several other grades underlie the floor of the pit.

of the embayment. These clays have attracted considerable attention in the last few years because of their use in glass pot and crucible manufacture. They belong to the refractory bond-clay type. Mining still continues but it is perhaps a little difficult to forecast the future of the field, since the deposits appear to lie in basins whose exact boundaries do not seem to be definitely known.

3. **The Piedmont and Mountain Belt.**—Lying west of the coastal plain, there is a broad belt extending from western Connecticut southwestward to northeastern Alabama. It is a belt in which new developments are taking place from time to time, but geologically speaking, all of the clays are of the residual type. That is to say, they have been derived from the weathering or breaking down of the rock to clay. These clays vary, then, according to the character of the original rock, which will affect their silica and iron contents.

FIG. 12.—Shaft for mining refractory bond clay north of Anna, Ill.

At the northeastern end of the belt, we have a somewhat isolated deposit of kaolin in western Connecticut.

The next region is in southeastern Pennsylvania where kaolins were formerly mined, but the deposits are now exhausted although the extension of the area in Delaware is still worked. No expansion can be looked for here. There are, however, two other districts in Pennsylvania deserving mention, not because they

are new discoveries, but because they contain possibilities. The first of these is a belt passing through Saylorsburg, Monroe County, and lying on the northwest side of Chestnut ridge. The clay here underlies the Oriskany sandstone. It has been used crude for portland cement, and in its washed form for paper. Recent developments give promise of its being of use in pottery and the belt should be further prospected.

It is interesting to note that white clays have been discovered in this same formation in northern West Virginia and northwestern Virginia, but at present they are not very accessible to transportation.

The second area is in the South Mountain district of Pennsylvania, where the mountain quartzite east and southwest of Mount Holly Springs, Cumberland County, has associated with it white residual clays. The clays are highly siliceous and the silica is very fine grained, so that the product needs careful washing. The crude clay is fairly white, but it does not burn so, so far as tests that have been made would indicate. The output, therefore, may be chiefly of value for paper and paints. Few companies are in operation here and the mining of the clay may present difficulties—due to the treacherous character of the ground—but the region shows possibilities for further development.

Our next area is in Virginia, where we have white residual clays on both sides of the Blue Ridge. Those on the eastern side are similar to the North Carolina ones, to be referred to later, but the Virginia deposits offer little promise—except one in the Nelson Co. district, in the neighborhood of the titanium ore mines.

Those on the western side form a chain of deposits extending southward along the flanks of the Blue Ridge on the eastern side of the Great Valley. They are usually associated with brown iron ore deposits and may be of considerable size. The clays which are derived from the weathering of shales, are white to grayish white when mined, but do not burn pure white in most cases. At present they are worked only at Cold Spring and Buena Vista, but there are other undeveloped deposits between these two points which are worthy of testing. The better grades are of value for paper manufacture and possibly ceramic products.

Considerable attention has been given to the occurrence of kaolin in western North Carolina, northern Georgia, and northeastern Alabama, where deposits occur, but it is only in the first-named state that the material seems to occur in any quantity, and while the qualities of this material seem to be fairly well known, the supply of future reserves is less in evidence. The present supply could be increased with sufficient coal and labor. Field work carried on in the region this summer shows also that

FIG. 13.—Excavation in white clay near Holly Springs, Pa. The overlying quartzite outcrops at the base of the slope seen in the distance.

the field contains considerable undeveloped material, although some of the deposits are a little remote from the railways.

With the reduction of imports, many questions are asked regarding the possibilities of finding kaolins in other parts of the country. The discoveries up to the present time are few. Indeed, in the northern states, where the soil has been combed off down to bed rock by glacial action, there is little chance of discovering any new ones. The deposits at West Cornwall, Con-

necticut, and St. Remi, Quebec, lying inside the glaciated area, are unique occurrences.

A district of white residual clay, long known but little developed, is in southeastern Missouri around Glen Allen and Lutesville. The deposits may be expected to differ in form from the North Carolina ones because of their slightly different derivation Those of North Carolina are formed from pegmatite veins and hence are long and narrow. Those of Missouri are derived from lime-

FIG. 14.—Pit in white residual clay near Cold Spring, Va. This clay is residual from Cambrian shale.

stone and hence may be extensive as to the clay mass. More work should be done in this area, but it still remains to be proven whether the deposits of white clay are of large extent. The bodies are irregular in shape and rest on limestone. It is a region of possibilities. The output at one mine is used in plaster board manufacture.

4. The Coal Measures Region.—In our central States, and along the mountains in some of our eastern States, coal fields of great

extent have been developed. Associated with these coals are extensive deposits of shale and clay, many of them being of refractory character and forming the basis of an extensive fire-brick industry, but here and there, refractory clays suitable for glass-pot manufacture have been developed. They are few and far between—the most noteworthy ones being those of the Cheltenham district around St. Louis. Others have been de-

FIG. 15.—General view of flint clay near Owensville, Mo.

veloped at scattered points in Illinois and Ohio, but one cannot help but feel that there is a possibility of finding more.

5. The Missouri Flint Clays.—There remains to be mentioned a peculiar type of clay found in Missouri and commonly referred to as flint clay, but this term hardly tells the whole story.

The deposits of this material are scattered over several counties, notably Gasconade, Franklin, Osage, Warren, etc., in the east-central part of the state. They are very numerous and while many have been opened up, we believe that numerous others

still remain to be found. A peculiarity is their frequent, basin-shaped form—due to the fact that the clay has been laid down in depressions. In these depressions we therefore find this more or less basin-shaped mass of refractory clay, often of the flint type, with its peculiar physical characteristics. Not the least remarkable feature is the mass of granular, öolitic clay, often found within the flint clay, and which runs high in alumina, even up to 75 per cent. This high alumina content is due pre-

FIG. 16.—Mass of disapore clay in flint clay deposit near Hofflin, Mo. The rod stands in front of a mass of diaspore clay. The man in foreground is standing on flint clay.

sumably to the mineral diaspore ($Al_2O_3.H_2O$). All of the deposits do not carry the rough clay, as the miners call it, and the quantity present does not seem to stand in any direct relation to the size of the deposit—for the masses may be large or small. The total tonnage visible at the present time is not large, but the total quantity which may be obtained by the continual opening of new pits may be considerable, and so it is a type well worth considering.

Conclusions.

In conclusion, we think it is safe to predict that the outlook for the still greater development of our domestic clays is good—assuming that labor and transportation facilities are available.

We can also assume, we believe, that the reserves in many of the areas referred to are also sufficient for some time to come, and lastly, that this country can supply satisfactory refractory clays of high bonding strength without the use of imported materials. It has also a considerable reserve of kaolins. A single clay may not have all the desired properties, but judicious blending of two or more clays may often yield a mixture having the desired properties.

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THE EFFECT OF CERTAIN IMPURITIES IN CAUSING MILKINESS IN OPTICAL GLASS.

By C. N. Fenner and J. B. Ferguson.

At the time when the staff of the Geophysical Laboratory began to coöperate with the Bausch & Lomb Optical Company in a study of the problems connected with the manufacture of optical glass (that is, in May, 1917) one of the matters which gave much concern, and, for a time, was probably the chief difficulty with which we had to contend, was the rather frequent production of pots of milky or opalescent glass, which was entirely useless for optical purposes.

In endeavoring to locate the source of the trouble and eliminate it, a very puzzling feature was the apparently random manner in which a pot of glass of this character appeared.

Among the four or five standard types of glass which were being produced in large quantities at the time, practically the only one which showed this phenomenon was the "light flint"—a glass which had a PbO content of about 33.5 per cent and whose refractive index was about 1.572.

Although the general procedure as regards the furnace treatment of each pot of this type was practically the same, pots of milky glass appeared in rather large quantities, while at the same time the greater number were perfectly clear.

The samples or proofs which were taken at intervals while the pot was in the furnace never showed any indication of milkiness, and frequently the trouble remained latent or nearly so even up to the time that the cold pot was taken out of the annealing arch, and did not show up until the apparently clear glass was reheated in the muffles for pressing. In fact, it might not be noticed until the pressed glass had come out of the cooling ovens. On the other hand in some instances, after the primary cooling of the pot in the arch the glass appeared milky throughout, or, more frequently, showed a slight opalescence at the top near the

circumference. In all cases in which there was any indication of milkiness reheating increased the turbidity, and even in those instances in which the opalescence appeared at first to affect but a small percentage of glass the whole potful was likely to become milky on reheating.

In endeavoring to obtain light on the matter we learned that this trouble had appeared quite recently, apparently about the time that the use of Russian potash instead of German in the batch had been begun, although information on the latter point was somewhat conflicting.

The German potash, of which there had been an abundant supply before the war and which had been used to the exclusion of potash from other sources, was a very pure material in every respect. The Russian potash contained variable but always considerable percentages of K_2SO_4 and KCl in addition to carbonate. The results of some analyses by Dr. R. H. Lombard of the Geophysical Laboratory are shown in Table I.

TABLE I.—ANALYSES OF RUSSIAN POTASH.

	SO_3.	Cl.	H_2O.	K_2CO_3.
1	0.75 per cent	..	..	..
2	1.9 per cent	7.02 per cent	..	..
3	...	3.6 per cent	..	..
4	2.47 per cent	2.03 per cent	..	..
5	12.2 per cent	3.4 per cent	10.1 per cent	56.1 per cent
6	7.7 per cent	3.6 per cent	7.5 per cent	68.7 per cent

In making up the batch about 68 parts by weight of potash (calculated to anhydrous K_2CO_3) to 300 parts of sand were used.

While the cause and prevention of the milkiness were still under discussion, Dr. H. E. Merwin drew our attention to an article which appeared in the *Journal of the Society of Glass Technology*,[1] in which very analogous phenomena were described. Their appearance was attributed to the presence of chlorides and sulphates (especially the latter) in Russian potash, and experiments in confirmation were carried out. Evidence was obtained

[1] J. D. Cauwood and W. E. S. Turner, "The Influence of Small Quantities of Chlorides and Sulphate in Producing Opalescence in Glass," *J. Soc. Glass Tech.*, 1, 187 (May, 1917).

by Cauwood and Turner that a high temperature and extended period of time in heating had beneficial results.

It seemed reasonable to suppose that our phenomena were to be accounted for in the same way, but certain considerations rather obscured matters. First: there did not seem to be any very direct connection between the amount of sulphate or chloride in the potash and the tendency of the glass to become milky. Second: a number of runs were made in an experimental furnace under conditions closely similar to those in the large furnaces. Groups of six small pots holding a few pounds of glass each were placed in the furnace simultaneously. Various amounts of sulphate and chloride were added to the batches. In a given group some pots showed milky glass and others did not, and here again there seemed to be no relation between milkiness and amount of sulphate or chloride added. Third: during a laboratory study of the ternary system CaO–MgO–SiO_2 by one of the present writers (J. B. F.) it had been found that with glasses having compositions within the SiO_2 field, especially with those not so high in SiO_2 as to bring the composition far from the two-phase boundary, a similar milkiness had often been obtained when the crucible was lifted from the furnace (above melting temperature) and quenched in water, especially if the transfer were not carried out very rapidly. At times the depth of milkiness was so great that the glass looked like the so-called Carrara glass. In such instances it had been possible to detect with the microscope multitudes of minute crystals of SiO_2. This phenomenon could not be ascribed to the presence of chloride or sulphate—as the "ingredients employed were" of a very high degree of purity. Therefore, it was evident that milky glass could arise from other causes.

The opinion toward which we leaned, in view of the evidence, was that sulphate and chloride were probably responsible in some way, but the remedy was not plain. It was not considered practicable to run the furnace temperature much above 1400° C, at which point it was supposed to be held at the time, without running much risk of corrosion and perhaps failure of the pots, nor was it possible to add carbonaceous material to these lead glasses to transform sulphates and chlorides to carbonates.

Nevertheless, before the cause of milkiness was definitely determined, a remedy was found, and later there was a gradual accumulation of evidence as to the cause.

In a previous paper[1] the senior writer has described the work which was done at the Bausch & Lomb plant preliminary to installing a means of temperature-control of a more reliable character than the thermoelements which had formerly been in use. This work showed that the readings given by the thermoelements varied widely from the true reading and, moreover, that the difference was not constant. Therefore, it became evident that the temperature treatment of different pots had been by no means as uniform as supposed. A Leeds and Northrup optical pyrometer was substituted and personal attention was given for several months to keeping the temperatures correct. The furnacemen were instructed in the use of the instrument, so that temperatures at night might be controlled by it, and in most cases dependence could be placed on instructions having been carried out. The result was that from that time forward for a long period there was scarcely a pot of milky glass produced, and when later a few appeared the reason was fairly obvious.

We took special pains to see that the temperature during melting and fining, that is, for a period of about 36 hours, was held at 1400° C or a little higher. The use of Russian potash was abandoned and in its place a much purer material supplied by the Armour Fertilizer Works was used. For the latter material specifications required that the SO_3 content of the potash should not exceed 0.3 per cent and that the Cl should not be in excess of 2.0 per cent. Analyses were made of the contents of each barre supplied and material exceeding these limits was subject to rejection. In general, the furnace temperatures were held at about 1410° C, in order to be on the safe side, and if the SO_3 or Cl content was near the limits considered safe, the temperature was run up to 1420° C. With this increased care and certainty of control, not only was the danger of milky glass al-

[1] C. N. Fenner, "Methods of Temperature-Control in Glass-Melting Furnaces," *Phys. Rev.*, 2nd series, 11, 2, 141. Abstract of a paper presented at the Rochester meeting of the American Physical Society, Oct., 1917.

most eliminated but also the loss of pots through leakage was very materially lessened.

For some time after the installation of this system, frequent tests were made of the tendency of the glass to become milky by heating small pieces for several hours in clay crucibles to a temperature of 950–1050° C in an electrical resistance furnace. It was found that in many instances glass which passed through all ordinary manufacturing processes without any indication of the appearance of milkiness still held sufficient latent possibilities in that direction so that the experimental treatment mentioned developed it to a point where it became visible. Study of the results showed in many cases that this development of opalescence or milkiness could be correlated with the fact that the potash used in the batch contained relatively large amounts of sulphate or that for some reason the furnace temperatures had been a lowed to drop below the established point for a considerable length of time.

At a period considerably later than that to which we have been referring it seemed desirable to cut down, if possible, the length of time for which pots remained in the furnace. Apparently as a direct consequence of this change pots of milky glass began to appear again. Whereas under the old schedules a potash containing 0.30 per cent SO_3 could be used without danger and one containing even 0.70 per cent SO_3 could be used without too much risk if the temperature were raised to 1420° or 1430° C, under the new schedule (about nine hours shorter) milkiness appeared when potash containing 0.30 per cent SO_3 was used in the batch. The trouble was much lessened, however, when the length of time of stirring was increased.

All the evidence leads to the inference that the trouble is to be ascribed to the presence of very small amounts of SO_3 and, to a less degree, Cl in the melt. Apparently these substances, when present, are continuously eliminated, but the evolution of the last traces is a very slow process. The elimination is favored by a high temperature and prolonged time, as Cauwood and Turner found, and also by the agitation of the melt which accompanies stirring.

If glass which is milky or is likely to become milky is heated to 1100–1150° C the milkiness disappears, but glass so treated develops numerous bubbles, which render it unfit for use, and a lesser degree of heating does not decrease the milkiness.

Several analyses (made by J. B. F.) show the actual SO_3-content of the glasses. In one case a very milky glass contained 0.14 per cent SO_3. After being cleared up by heat-treatment at 1100° C or a little higher, and having thereby lost its tendency to become milky, the same glass contained 0.06 per cent SO_3.

In another case two lots of glass were made at the same time in the same furnace (a two-pot furnace) and from the same batch. Probably as a result of unequal distribution of temperature in the furnace, one pot became milky and the other did not. The glass from the first pot was found to contain:

SO_3	0.10 per cent
Cl	0.17 per cent

The glass from the second contained:

SO_3	0.05 per cent
Cl	0.17 per cent

Probably n general the presence of Cl in the potash is less serious than the presence of SO_3 when the heat-treatment is similar to that which we followed, but at times enough Cl may be left to strengthen the effect of SO_3 and cause milkiness to appear in a glass which would otherwise remain clear. The last case may be an example of this.

Although the origin of milkiness in glass in the cases described seems to be connected fairly definitely with the presence of SO_3 or Cl, it is doubtful whether the minute particles to which the milkiness is due are themselves composed of sulphates or chlorides. Direct evidence on this point is very difficult to obtain. With the best microscopic magnification it is barely possible to discern discrete particles of foreign matter and nothing can be made out regarding their properties. In fact, the bluish color of the opalescence is itself evidence that the particles are so minute as to be of a similar order of magnitude to the wave-length of light and thus produce a scattering of light rays. According to Wood[1]

[1] R. W. Wood, "Physical Optics," 1911, p. 624.

"If a beam of light is passed through a transparent medium containing in suspension small particles, the refractive index of which differs from that of the surrounding medium, light will be given off by the particles in all directions. In the case of particles of the order of magnitude of the light waves, the amount of light scattered increases as the wave-length is decreased, which explains the preponderance of blue always observed in these cases." However, it was found by Mr. H. S. Roberts, of the Geophysical Laboratory, that the size of the particles may be increased by holding a piece of glass of this character at a softening temperature (say 900–1000° C) for several days. Dr. H. E. Merwin examined microscopically the glass so treated and was able to determine that the particles now showed an angular form and possessed a refractive index considerably below that of the glass in which they were imbedded. It seems most probable that the action of the sulphate and chloride impurities is of the sort to which the term "catalytic action" is applied for lack of better knowledge. Ordinary glasses are undoubtedly in a state of unstable chemical equilibrium at the temperature at which the final stages, at least, of the furnace work is carried on. Their stable condition would be one of partial devitrification, and it is an astonishing fact that the unstable state is retained for a prolonged period under conditions of treatment which seem favorable to crystallization. Whatever may be the factors which cause the glassy condition to be retained it appears that the presence of sulphates and chlorides, in quantities so minute that the physical properties should apparently be in no way affected, nevertheless does cause such a change as to permit swarms of sub-microscopic crystals to develop. The phenomena exhibited are of a kind for which very little real explanation can be offered. In geological processes it has long been recognized that the presence of certain volatile constituents, termed mineralizers, is especially favorable to the segregation and crystalline development of minerals in rocks, and the matters here described are apparently of a closely similar nature.

Another occurrence at the Bausch & Lomb plant is of interest in this connection. The light crown glass which was made at the works was never known to be affected with milkiness and it was

supposed that the Russian potash on hand could be employed without detriment in making glass of this type. In a case in which this was attempted the potash contained 6.5 per cent SO_3, 3.6 per cent Cl and 7.5 per cent H_2O. During melting the impurities separated out and formed a layer of "salt water" on the surface of the glass. At a later stage, during cooling, the glass became so viscous that stirring had to be stopped and the pot withdrawn. When cold the resultant glass was found to contain quantities of nodules or spherulites of devitrified material, whose general diameter was four or five millimeters, although some masses of much larger size were present. Microscopic examination of this material shows that it has a refractive index of about 1.485, very low birefringence, and the typical branching skeleton forms of cristobalite—the high temperature form of silica.[1] A determination by Dr. R. H. Lombard gave a content of 0.28 per cent SO_3 in the glass, and the conditions seemed to be favorable in this instance for the glass to retain the maximum amount of SO_3 possible. The small amount present acted as a very efficient aid to devitrification.

An analysis made by Dr. E. T. Allen on a glass for searchlight mirrors manufactured in England, gave 0.74 per cent SO_3 and 0.10 per cent Cl. The other important constituents were

	Per cent
SiO_2	71.34
CaO	14.73
Na_2O	10.37
K_2O	0.44

This glass showed no indication of milkiness or devitrification. This fact and other evidence given show that the devitrifying effect of sulphate and chloride varies with the composition of the glass.

Although in the cases which have been described the evidence indicates that SO_3 and Cl were responsible for the production of milky glass, it is quite certain that these are not the only substances which are likely to give rise to it. The peculiar "mineralizing" rôle played by SO_3 and Cl is probably filled in some cases

[1] C. N. Fenner, "Stability Relations of the Silica Minerals," *Am. J. Sci.*, [4] **36**, 355 (Oct., 1913).

by As_2O_3 or CaF_2, and there may be other substances having like effect. Also some types of glass (probably quite different in chemical composition from ordinary commercial glasses), such as the CaO-MgO-SiO_2 glasses mentioned on an earlier page, have a tendency toward devitrification in this opalescent or cloudy form, which is apparently inherent in them and does not arise from impurities.

Summary.

In the manufacture of optical glass at the Bausch and Lomb plant, a matter which gave considerable difficulty for a while was the occasional production of pots of glass which were affected by opalescence or milkiness. The evidence indicated that the source of the trouble lay in the sulphate and chloride content of the Russian potash to which recourse was had when the German supply was cut off, although certain facts tended to cast doubt upon this conclusion. It was found, however, that the trouble disappeared when more reliable methods of temperature control were installed, by which an assurance could be had of keeping the temperatures constantly at 1400–1420° C, and when the Russian potash was replaced by an American product, more nearly free of impurities. Later, evidence was obtained which connected the milkiness quite definitely with the impurities mentioned, at least as regards the case under discussion, although in other cases the same effect is to be ascribed to other causes.

Reasons are given for the conclusion that the milkiness is caused not by the separation of sulphates or chlorides themselves but to some slight change in the physical properties of the melt which permits the separation of clouds of minute crystals of cristobalite.

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September 25, 1918.

SILICA REFRACTORIES.[1]

By Donald W. Ross, Pittsburgh, Pa.

Previous Work.

A great deal of work has been done on the stability relations of the silica minerals, the results of which are probably summarized best by Fenner.[2] More recently, the results of these studies have been applied in the optical examination of silica brick. McDowell[3] has been one of the foremost investigators along this line, and at the present time Insley[4] is conducting an exhaustive study of silica brick made from practically all of the leading varieties of materials used commercially in the United States for this purpose.

The present status of our knowledge along these lines is briefly as follows: The specific gravity of quartz is 2.65, that of cristobalite is 2.33, and that of tridymite, 2.27. Upon heating, alpha-quartz (the stable form at atmospheric temperatures) is inverted to beta-quartz at 575° C. In the presence of a flux, this beta-quartz is transformed into beta-tridymite at 870° C. This, in turn, is transformed at 1470° C into beta-cristobalite—which is the stable form from this temperature to its melting point, 1715° C. "In the absence of a flux, the beta-quartz is transformed directly to cristobalite—which in this case is the final form."[5] In the manufacture of silica brick two per cent of lime (CaO) is mixed with the quartzite. Upon heating, the lime

[1] By permission of the Director, Bureau of Standards.

[2] C. N. Fenner, "The Stability Relations of the Silica Minerals," *Am. J. Sci.*, **36**, 331-384 (1913). In this connection it is of interest to note that work recently published by the Geophysical Laboratory of the Carnegie Institution, indicates that the melting point of pure cristobalite is probably 1715° ± 10° C instead of 1625° C as previously reported.

[3] J. Spotts McDowell, "A Study of the Silica Refractories," American Institute of Mining Engineers, *Bull.* **119**, p. 1916.

[4] H. Insley, U. S. Bureau of Standards, Pittsburgh, Pa.

[5] *Am. J. Sci.*, **36**, 339 (1913).

combines with a small amount of the silica to form a glass, or, in some cases, possibly a little mono-calcium silicate ($CaO.SiO_2$). The quartz of a silica brick is transformed first to cristobalite, but as the lime constitutes a small amount of flux, continued heating causes the silica to be slowly converted into tridymite—after approximately two-thirds of it has been transformed into cristobalite.

McDowell[1], Bell,[2] Le Chatelier[3] and others have also studied the differences in finished silica wares due to the use of the different quartzites and to variations in the methods of manufacture.

Usual Method of Manufacture.

Ordinarily, silica brick are manufactured by crushing the quartzite so that it will pass through a 2-inch ring. The crushed material is then fed in batches into wet pans and ground until the largest particles are approximately 0.2 inch in diameter, during which period two per cent by weight of CaO as milk of lime is added along with enough water so that the resulting mix can be molded into bricks. When dry, the bricks are burned in fire-brick kilns of the usual type.

Nature of the Present Work.

Some three years ago, the writer began a study of the silica refractories under the direction of Mr. A. V. Bleininger. The present paper is an attempt to set forth a summary of the data obtained since that time and to present some applications of the same. In taking up the data we shall first consider the raw materials, then manufacture and burning, and lastly some of the properties of the burned ware.

Raw Materials.

By far the largest part of the silica brick in all countries are manufactured from quartzites of comparatively early geologic age, although in some cases, chalcedony, chert, quartz sand, or pebbles cemented together with chalcedony, have also been

[1] *Loc. cit.*

[2] C. E. Nesbitt and M. L. Bell, "Silica Refractories," *Proc. Am. Soc. Testing Materials*, 1917.

[3] *Rev. métal.*, June, 1917.

used. Suitable quartzites range from the hard, highly metamorphosed varieties having tightly interlocking grains, to the medium soft rocks which are slightly porous. Loose grained quartzites and sand stones are, however, of little value. The quartzites now being used range from the very young rocks to the oldest sedimentaries.

The following microscopic method may prove of value in judging the suitability of quartzites for the manufacture of silica brick. In the metamorphosis of sandstones to quartzite, the rocks which received but little alteration consist of hard, usually rounded grains of quartz imbedded in a ground mass of a softer material. If a thin section of a rock of this kind is viewed under the microscope, the softer material appears as cloudy areas between the quartz grains. These cloudy areas consist of fragments of silica (probably quartz) and impurities such as hydrated iron oxide. As the metamorphosis proceeded, the interstitial silica, frequently augmented by silica from water solution, crystallized on the larger grains and shows the same orientation as these grains. Silica crystallizing in this way is pure—except for occasional inclusions. Hence, the impurities are segregated. In thin sections of highly metamorphosed quartzites these segregations appear as sharply defined lines between the tightly interlocking, built-up grains of quartz. The quartzites which have been found most satisfactory for the manufacture of silica refractories are those which have moderately to tightly interlocking grains, while those materials which merely show cloudy areas between the harder grains have been found to be less desirable. The specific gravity of quartz is 2.65. The specific gravity of chert rock—although practically the same as that of quartz—is usually slightly below this figure as shown by No. 29 (Table 1) (2.585). This is borne out by Mellor.[1]

Screen analyses (by the wet method) and porosity determinations were made on 15 leading varieties of unburned silica brick. These are presented in Table 2, together with the porosities and specific gravities of bricks of the same varieties which have received regular commercial burning.

[1] J. W. Mellor, *Trans. Eng. Ceram. Soc.*, **15**.

TABLE 1.

Materials.					Micro-Composition.		
Geological formation from which derived.	Remarks.	Sample No.	Specific gravity.	Per cent porosity.	Quartz plus silicates.	Cristobalite.	Tridymite.
Medina	Commercial brick, reheated for 11 days; 4 days at 1450° C and finally 1 day at 1500° C.	33	2.321	29.45	Largely cristobalite and tridymite.		
	Commercial brick, reheated 40 times to 1450° C on bag wall of test kiln.	34	2.275	31.85	Small amounts of silicates and cristobalite. Rest is tridymite.		
	Commercial brick, reheated 10 times in commercial silica brick kiln to cone 14.	35	2.271	27.25	11	28	61
	Commercial mix, heated 9 times at successively higher temperatures from 1200–1500° C by 50° intervals—last 3 heatings were to 1500° C.	36	2.304	25.19	6	70	24
Baraboo	Commercial brick which received 9 years' service in Koppers by-product coke oven. Micro-analysis 1 is from the side exposed to heating flue; 2 from center of brick; and 3 from coke chamber side.	37	2.314	19.94	No. 1: 8 / 2: 20 / 3: 31	3 / 13 / 24	89 / 67 / 45
Homewood	Raw rock.	38	2.626	6.52			
Chert from Mitchell limestone, Indiana.	Raw rock.	39	2.585	1.83			
	Rock calcined rapidly (6 hours) to cone 20.	40	2.272	14.33			
	Commercial brick made from chert rock.	29	2.273	25.64	5	12	83
	Commercial German brick, Stella Werke (gives good service).	45	2.372	20.07	20	78	2
	Test brick, California quartz + river sand + 2% CaO, 2 heatings in commercial clay brick kiln.	41	2.437	30.02			
	Alabama mica quartz schist rock.	42	2.690	4.52			
	Test brick made from above schist.	43	2.616	34.07			
	Test brick made from slightly impure silica sand from Georgia + 8% $Ca(OH)_2$.	44	2.527	30.40			

TABLE 2.

Screen analyses (by the wet method) of raw commercial silica brick mixes. The porosities of the raw and burned bricks and the specific gravities of the burned bricks of the same brands are also shown.

TABLE 2.

Screen analyses (by the wet method) of raw commercial silica brick mixes. The porosities of the raw and burned bricks and the specific gravities of the burned bricks of the same brands are also shown.

Screen. No. mesh.		Medina.						Baraboo.		Homewood Formation.	
	No. of brick.	6	2	7	3	5	4	18	21	14	16
	% on screen.	%	%	%	%	%	%	%	%	%	%
4	..	1.69	2.44	1.43	1.27	9.97	1.41	3.58	1.72	3.25	11.96
8	..	19.85	24.18	20.22	14.35	15.63	23.46	27.38	20.98	19.09	11.96
20	..	11.74	9.67	9.15	10.79	10.01	16.07	15.66	17.86	6.09	6.20
30	..	3.31	6.36	9.15	9.84	7.76	3.75	5.77	8.84	1.89	4.95
40	..	10.07	9.67	9.69	15.34	17.23	10.93	4.90	7.97	10.22	29.62
60	..	18.65	13.97	17.13	16.48	12.17	15.02	12.20	5.73	26.35	16.94
80	..	1.58	5.70	4.99	3.85	2.97	3.60	2.23	4.24	6.29	2.81
100	..	3.17	4.26	3.76	3.46	3.27	5.68	3.97	3.32	2.07	5.55
150	..	4.25	3.33	4.10	2.94	2.32	3.91	3.14	2.60	3.87	1.83
200	..	3.35	2.45	3.07	2.47	1.51	2.90	2.77	2.76	2.55	1.78
Through 200	..	9.46	6.63	6.25	5.84	2.88	4.39	8.57	11.27	6.89	8.48
Floated	..	12.84	11.34	11.06	13.37	14.28	8.90	9.84	12.80	11.38	9.89
Porosity, raw brick		26.70	26.15	25.93	27.20	21.22	25.45	23.54	23.20	27.50	28.54
Porosity burned brick		26.35	25.84	28.35	28.52	22.80	27.16	23.91	23.45	30.58	26.55
Sp. gr., burned brick		2.291	2.295	2.375	2.424	2.340	2.393	2.381	2.390	2.496	2.517

Screen. No. mesh.		Colorado.		Mo.	Chert.	Eastern Penna.	Average of all varieties.	Theoretical[1] for minimum porosity.[1]	Diameter of holes between wires. Inches.
	No. of brick.	28	27	31	29	12			
	% on screen.	%	%	%	%	%	%	%	
4	..	0.23	3.27	0.54	0.37	0.39	2.24	0.40	0.18
8	..	14.17	16.18	11.09	15.47	21.76	18.85	28.10	0.093
20	..	16.55	22.53	18.15	25.00	22.04	14.52	28.20	0.034
30	..	3.25	12.58	9.90	12.14	6.31	7.06	10.30	0.0198
40	..	4.09	10.06	9.80	4.77	6.45	10.72	4.40	0.0150
60	..	12.05	7.09	11.87	7.55	11.57	13.66	6.10	0.0087
80	..	3.62	1.95	3.43	2.37	3.03	3.51	2.50	0.0068
100	..	3.08	4.10	4.09	3.93	3.81	3.83	2.50	0 0055
150	..	3.92	4.40	3.16	2.78	3.32	3.32	2.25	0.0041
200	..	5.04	4.21	3.03	2.48	2.97	2.89	1.75	0.0029
Through 200	..	21.32	4.09	9.57	11.44	9.83	8.46 }	13.50	..
Floated	..	12.65	9.53	15.37	11.66	8.53	11.56 }		
Porosity, raw brick.....		26.52	22.93	25.30	24.70	20.32	...	...	..
Porosity, burned brick.		24.51	24.63	24.70	25.64	23.10	...	...	..
Sp. gr., burned brick...		2.387	2.393	2.393	2.273	2.480	...	...	..

[1] Taylor and Thompson, "Concrete, Plain and Reinforced," p. 775. Formula for construction of curve is $d = P^2D/10000$, where
D = Diameter of largest grain.
d = Any given diameter.
P = The per cent of mixture smaller than any given diameter.
Largest grain = 0.182 inch diameter.

Curves showing the results of screen analyses made on quartzites that have been crushed ready to be molded into brick, when compared with the theoretical curve for minimum pore space,[1] show that there is always an excess of material of the sizes which correspond to those of the quartz grains.

In crushing, sandstones and quartzites that are but slightly metamorphosed break mostly to individual sand grains and yield an excess of these sizes.

Moderately metamorphosed material, *i. e.*, material that is still slightly porous, follows the theoretical curve quite closely. In this case the breaking is usually through the interstitial material between the built-up grains. However, the interstitial material breaks free from some of the original rounded grains and still other grains are broken in two.

In the case of highly metamorphosed quartzites, *i. e.*, those in which the crystalline quartz is practically continuous, the breaking is usually through the grains, but a fair percentage of rounded grains also break free from the interstitial material. This breaking away of the interstitial material from the rounded grains is particularly noticeable in the highly metamorphosed Baraboo quartzites.

Inspection of the screened material revealed the fact that in the Medina, Baraboo, Homewood, Missouri, and Eastern Pennsylvania materials, the original rounded grains which broke free from the interstitial material practically all passed through a 30-mesh (0.503 mm. diam.) screen, and that but very few particles passed through a 60-mesh (0.221 mm. diam.) screen—the great majority being caught on the 40-mesh (0.381 mm. diam.) and 60-mesh screens, in varying proportions. In Fig. 1 are shown photographs of samples of material remaining on the 40-mesh screen. No. 6 consists largely of material broken through the interstitial material between the built-up grains, and some that has broken through the grains. A slight amount of finely ground material still adheres to the particles. No. 21 shows a large percentage of the original rounded grains which have broken free from the material of the built-up grains. No. 12 contains a large percentage of material broken through the grains and

[1] Taylor and Thompson, "Concrete, Plain and Reinforced," p. 775.

also material in which the breaking was between the original rounded grains and built-up grains. A slight amount of finely ground material still adheres to the particles. In No. 14, the original grains of the material appear more angular than is the case with the other quartzites examined. In No. 31, many

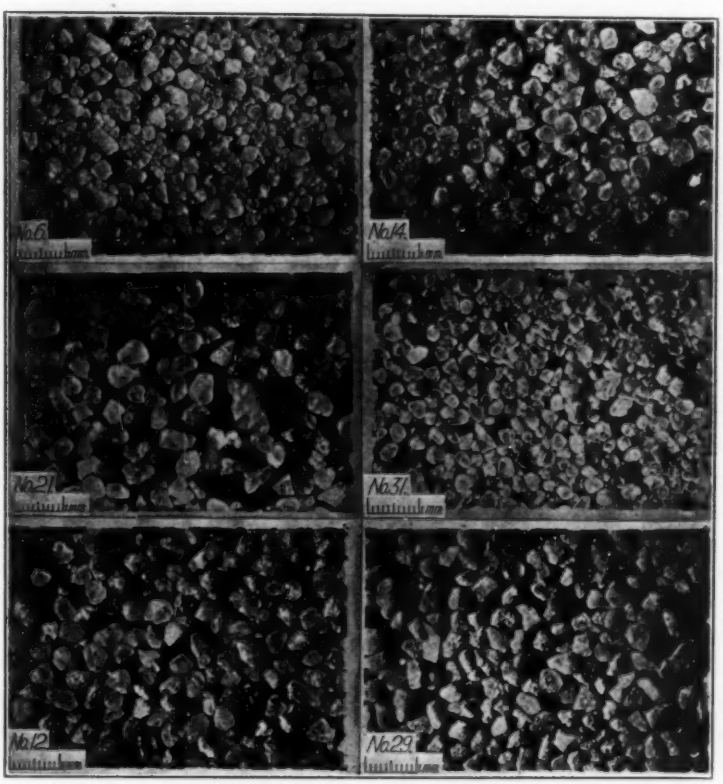

FIG. 1.—Materials (caught on 40-mesh sieve) resulting from wet screen analyses of raw commercial silica brick mixes. Magnified. (Microphotographs by Dr. R. Thiessen, U. S. Bureau of Mines, Pittsburgh, Pa.)

of the original rounded grains have broken free from the chalcedony bonding material. In No. 29, the grains in the chert are extremely fine, hence, as might be expected, it breaks into angular fragments. A slight amount of finely ground material

still adheres to the particles. The three grains checked are of a different material which was accidentally included.

At the present time, McDowell is carrying on a study of the sizes of the built-up interlocking grains of several quartzites.

Of the quartzites, the loosely bonded varieties yield the most porous raw brick, and the strongly bonded varieties, in which the breaking is across the grains—thus avoiding an excess of material corresponding to the grain sizes—yield raw brick of the least porosity. Material No. 12 (Table 2) is of this type and has the lowest porosity of any of the unburned brick tested. The chert material, No. 29, is next, this being closely followed by the mixes made from Baraboo quartzite, while the large majority of the raw mixes made from the Medina quartzite, of Blair and Huntingdon counties, Pennsylvania, show in turn somewhat greater porosities than do the Baraboo materials.

Innovations in Methods of Manufacture.

Finished silica brick frequently show quite wide variations in their physical properties when subjected to strength tests, etc. These variations may be due in part to differences in grind from one pan to the next, and, in part, to the human factor in hand molding. To overcome such difficulties in special cases, the following changes in the methods of manufacture have been suggested:

The material is ground and, by screening, is divided into three sizes. These are then mixed in the desired proportions in a machine similar to that used for mixing concrete. To avoid storage in bins,[1] the material might be ground directly in a wet pan having raised mullers. The lime is added at this point. The brick are then machine molded. This should insure greater uniformity of texture, shape, amount of material per brick, and should tend to slightly reduce the porosity. Nesbitt and Bell[2]

[1] When crushed materials are poured from a spout into a bin there is a tendency for the coarse material to segregate from the fines. This trouble may be largely obviated by frequently changing the position of the spout while a bin is being filled. It is essential to keep this in mind when using bins for the storage of ground silica brick materials.

[2] *Loc. cit.*

have suggested that 1500 pounds per square inch is the most desirable pressure for use in the manufacture of machine molded silica brick. Fine surface cracks appear to be characteristic of machine-pressed silica brick. However, in most cases these should be of no serious detriment to the bricks. In making silica brick by machine, the water content need not be quite so great as for the hand-made brick. However, an abnormally low water content is apt to result in an open brick of weak structure—both in the raw and burned condition.

Effects Produced by Burning.

Since the specific gravities of cristobalite (2.33) and tridymite (2.27) are considerably lower than that of quartz (2.65), the relative position of the specific gravity of any sample of silica brick between these limits should indicate, quite accurately, the degree to which its quartz has been transformed to the lower specific-gravity forms.

To accurately determine the specific gravities of solids by means of a pycrometer bottle, using finely powdered material, is a rather tedious operation. Hence, an effort was made to determine the specific gravity by using a two-inch cube of the material. This was first weighed dry, then saturated with water and weighed wet, and finally the saturated piece was weighed, suspended in water. From these data, the specific gravity was calculated. Saturation was obtained by placing the pieces in boiling water and subjecting them, while thus submerged, to a vacuum equivalent to twenty-four inches of mercury for a period of four hours. This method, when carefully conducted, has been found to give the specific gravities with sufficient accuracy for our present purpose.

The specific gravities of most of the leading varieties of commercial silica brick, manufactured in the United States, have been determined in this way (see Table 3) and, thus far, all lie between 2.65 (the specific gravity of quartz) and 2.27 (the specific gravity of tridymite). The results of our own work, and more recently of that of other investigators of silica refractories, indicate that this gives a definite and satisfactory indication of the degree to which a brick has been burned.

TABLE 3.—POROSITIES AND SPECIFIC GRAVITIES OF PRINCIPAL BRANDS OF COMMERCIAL SILICA BRICK MADE IN THE UNITED STATES.

Quartzite from which the brick were made.	Sample No.	Specific gravity.	Per cent porosity.
Medina (Tuscarora)	1	2.375	27.15
	2	2.296	25.84
	3	2.424	28.52
	4	2.393	27.16
	5	2.340	22.80
	6	2.291	26.35
	7	2.375	28.35
	8	2.336	31.52
	9	2.321	25.97
	10	2.358	26.10
	11	2.291	29.56
	12	2.460	23.10
	13	2.340	29.88
		Av., 2.357	Av., 27.07
Homewood sandstone	14	2.496	30.58
	15	2.448	28.95
	16	2.517	26.55
		Av., 2.470	Av., 28.69
Oneida	17	2.274	25.66
Baraboo	18	2.381	23.91
	19	2.430	25.15
	20*	2.325	31.41
	21	2.390	23.45
	22	2.395	24.79
		Av., 2.399	Av., 24.30
Quadrant formation (Montana)	23	2.495	23.22
	24	2.490	22.66
	25	2.537	22.96
Alabama, probably Weisner formation.	26	2.311	29.75
Dakota and Comanchian (Colorado)	27	2.393	24.63
	28	2.387	24.51
Indiana chert	29	2.273	25.64
Saint Louis district	30	2.363	25.73
	31	2.393	24.70
Eastern Pennsylvania	32	2.335	26.85
Grand average of all varieties		Av., 2.384	Av., 26.34

* Calcined quartzite.

Using the specific gravities of the materials in this way, our data (Table 4 and Fig. 2) indicate that at the end of heating to 1500° C (conducted as follows: heating to 800° C in 18 hours, heating from 800° C to 1500° C in 6 hours, and held at 1500° C

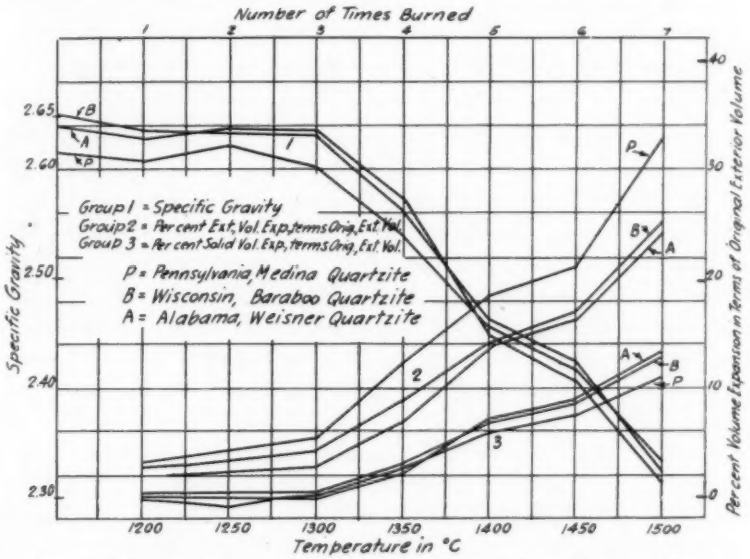

FIG. 2.—Changes in exterior and solid volumes and specific gravities of Medina, Baraboo and Alabama quartzites, caused by repeated burnings to successively higher temperatures from 1200°–1500° C.

for $1^1/_2$ hours) the amount of transformation that had taken place was approximately the same in the Baraboo, Alabama, and Medina quartzites. The specific-gravity curves show that, for the repeated heatings at from 1300° to 1400° C, the Medina quartzite appears to be transformed slightly more than the other two. The average specific gravity (Table 3) of commercial brick made from the Baraboo quartzite is somewhat higher than that of brick made from the Medina quartzite. Hence, since the chief part of the burning received by average commercial brick is between 1300° and 1400° C, it is probable that at these temperatures Baraboo quartzite is actually transformed at a slightly slower rate than is the Medina

TABLE 4.—SPECIFIC GRAVITIES OF QUARTZITES BEFORE AND AFTER HEATING FOR $1^1/_2$ HOURS AT 1500° C.

Material.	Specific gravity. Raw quartzite.	Specific gravity of quartzite after 1500° C burn.
Medina quartzite	2.635	2.303
Baraboo quartzite	2.650	2.293
Alabama quartzite	2.640	2.295
Homewood sandstone	2.630	2.326
Montana quartzite	2.638	2.306

quartzite. When quartz is transformed to cristobalite, the action is progressively from the surface and along cracks towards the interior and hence is most rapid in the varieties having the greatest surface areas. In accordance with this we would expect the slightly porous Medina quartzite to be transformed somewhat more rapidly than the more highly metamorphosed, non-porous, Baraboo quartzite. At temperatures above 1400° C, however, the transformation of quartz to the lower specific-gravity forms is so rapid in all varieties that the above effect is apparently obscured.

In silica brick, the transformation from cristobalite to tridymite apparently takes place first in the fine material of the ground mass and then progressively from the surfaces to the centers of the larger particles. This is illustrated in Fig. 3, which shows a

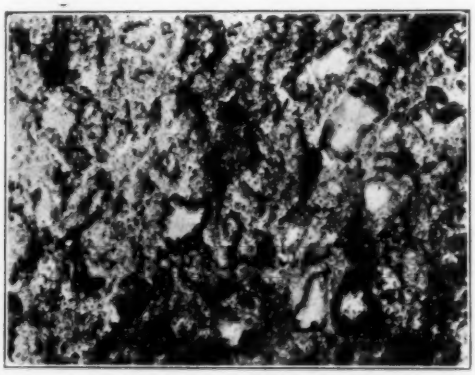

FIG. 3.—Brick made from Medina quartzite, after 40 heatings to 1450° C. Ground mass is tridymite, gray outer portion of large particles is tridymite in glass, and white centers of large particles are cristobalite (natural size).

fractured surface of a brick which has been burned to an advanced stage. In this case the quartz has almost entirely disappeared and practically all of the cristobalite that remains is represented by the light patches present at the centers of the larger particles. In the course of the transformation of quartz through cristobalite to tridymite, the original crystal form is largely lost, so that the final product shows a considerable quantity of interlocking crystals which were not present in the unburned brick.

In accordance with Mellor,[1] we found that chert is transformed to the lower specific-gravity forms much more rapidly than quartz. Thus, brick 29 (Table 3) (micro-analyses shown in Table 1), which was made from a chert rock, received one regular burn in a commercial silica brick kiln. Its specific gravity is lower and the sum of its contents of cristobalite and tridymite is much higher than is usually the case with single burn bricks made from quartzite.

A comparison of the porosities of raw and burned brick (Table 2) shows that the porosity of the burned brick depends largely upon the porosity of the raw mix. However, it was found, by repeated trials, that if a raw brick mix was heated to 1500° C in one day the resultant product was porous, weak and friable, while if fourteen days were consumed in reaching the same maximum temperature, with a protracted soaking at from 1200° to 1350° C, the resultant brick showed no undue increase in porosity and was sound and strong. In fact, in cases where the conversion of quartz has been practically completed at the soaking temperatures, the final increasing of the temperature to the maximum actually causes the porosity of the brick to decrease. As a rule, the increased porosity (punkiness) of rapidly burned brick is much more pronounced with loosely than with firmly bonded quartzites.

Number 37 (Table 1) represents a piece of brick which received long soaking in use in a by-product coke oven at moderately high temperatures. The differences in micro-composition from the hot (heating flue) side of the brick to the cold (coking chamber) side are very marked. For the specific gravity and porosity determinations, pieces were taken which extended entirely across

[1] *Loc. cit.*

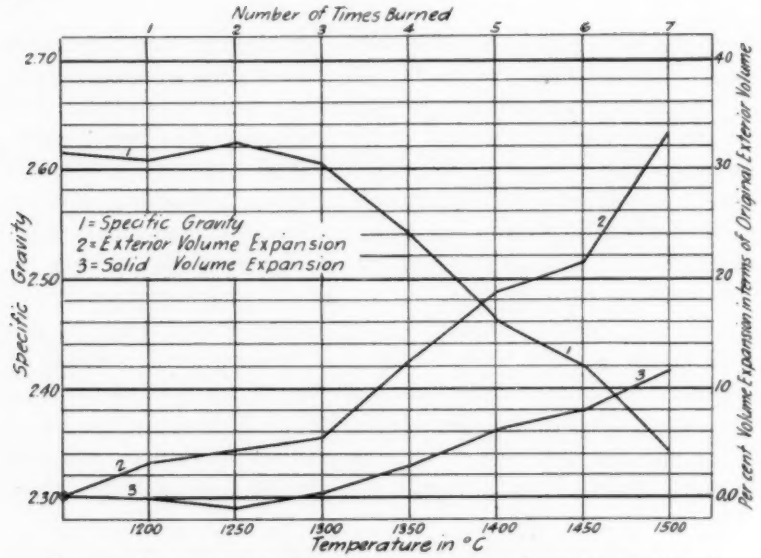

FIG. 4.—Changes in exterior and solid volume and specific gravity of Medina quartzite, caused by repeated burnings to successively higher temperatures from 1200°–1500° C.

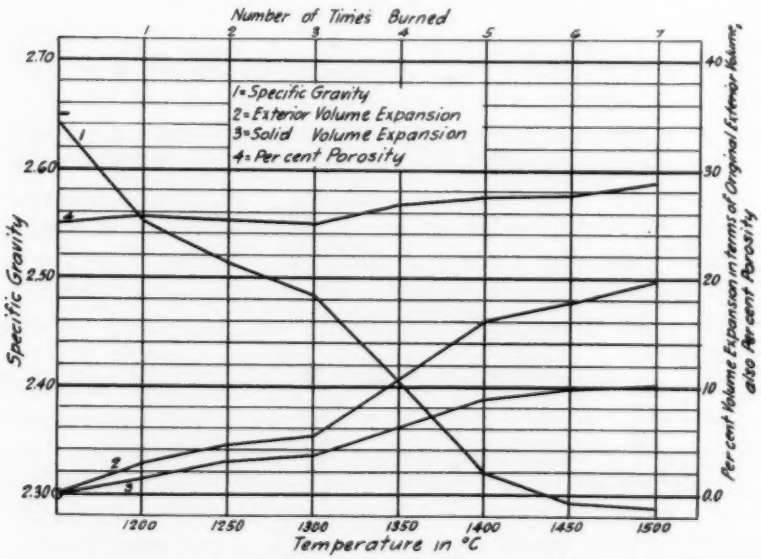

FIG. 5.—Changes in exterior and solid volume, specific gravity and porosity of Medina raw mix—caused by repeated burnings to successively higher temperatures from 1200°–1500° C.

a section of this brick. The abnormally low porosity of this brick was in all probability caused by the protracted soaking at high temperatures which it received in use. This brick is of particular interest because of its low cristobalite content, even atthe coke side—where there is considerable quartz + silicates present.

A comparison of the specific-gravity curves in Figs. 4 and 5 indicates the way in which the lime increases the amount of transformation produced by a given heat treatment. Increasing the lime content above two per cent appears to further increase the rate of transformation. Sodium chloride apparently has a similar effect. On the other hand, plastic clay, pure calcined alumina (ground to pass through a 60-mesh screen), and iron oxide appear to have very little effect on the rate of transformation. Small percentages of fluorspar (CaF_2) were tried in conjunction with lime, and although our data is not conclusive, the indications are that the fluorspar has very little effect on the rate of transformation.

Properties of the Burned Brick.

The results of cold cross breaking tests (Table 5),[1] in conjunction with the natures of the materials broken, and their specific gravities, porosities (Table 3), micro-analyses, etc., indicate that, with brick made from quartzites, the strength of slightly to moderately burned brick probably depends largely upon the

TABLE 5.—CROSS BREAKING TESTS. (MODULUS OF RUPTURE.)

	Material.				
Test No.	(18) Baraboo.	(6) Medina.	W. Va.	(29) Chert.	Medina.[2]
1	565	1557	664	446	...
2	593	709	1143	...	...
3	544	452	782	...	...
4	546	1003	645	...	...
5	527	863	809	...	...
Av.	555	915	809	...	793

[1] Bricks set on edge with six inches between supports.

[2] As given by McDowell.

state of metamorphosis of the raw material and the porosity of the raw brick and the lime-silica bond formed in the burning. On the other hand, well burned silica brick appear to have their strength somewhat increased by the formation of interlocking crystals—occasioned by the transformation of the quartz to the lower specific-gravity forms. Seaver[1] likewise maintained that the interlocking crystals of well-burned silica bricks increases their strength.

If all other properties of a silica brick made from quartzite are found satisfactory, it is not usually found that the brick will fail in use because of lack of strength. The Baraboo and Medina varieties (Table 5) have both been thoroughly tried out in steel furnace and coke oven work and both give consistently satisfactory results. The great variations in strength of the Medina brick, as compared with the Baraboo, may be due to an unequal development of interlocking crystals in the individual specimens of the former.

In Table 6 is presented the data of crushing tests made upon "Star" silica brick at various temperatures as reported by

TABLE 6.—CRUSHING STRENGTH OF SILICA BRICK AT VARIOUS TEMPERATURES (LE CHATELIER).

	Crushing strength of hot brick.	
Temperature ° C.	Kg./sq. cm.	Lbs./sq. in.
15	170	2418
520	158	2247
670	150	2133
806	139	1977
950	125	1778
1050	120	1707
1200	85	1209
1320	62	882
1460	50	711
1540	37	526
1600	30	427
1700	12	171[2]

[1] K. Seaver, "Manufacture and Tests of Silica Brick for the By-Product Coke Oven," *Trans. Am. Inst. Mining Eng.*, **53**, 125–139 (1916).

[2] Extrapolated.

Le Chatelier.[1] The 1700° C figure has been extrapolated by a continuation of the curve. This curve would seem to indicate that hot crushing tests, to be of the most value, should be made at a temperature that corresponds to the working conditions—such as the temperatures of the crowns of steel furnaces. Or, if made at any other temperature, the strength obtained should be compared to that at the corresponding temperatures on previously prepared curves of the above nature. Such prepared curves should of course include varieties of materials similar to those under test.

McDowell,[1] in his tests of silica brick which had been burned from one to ten times in a commercial kiln, found that at the end of the first few burns the strength had reached a maximum—after which it declined slightly. His theory is that this decrease is due to a slight rupture of the brick—caused by repeated heating and cooling—rather than to an inherent weakness of one or both of the low specific-gravity forms of silica.

In making load tests on silica brick, it was soon found that, if heated to 700° C at the same rate as clay brick (270° C in 20 minutes, 520° C in 40 minutes, and 670° C in 60 minutes), the specimens invariably spalled; and that spalling apparently ceased when the rate of heating below 500° C was decreased to 50° C in 15 minutes. Measurements of the linear expansion of bricks during the load tests indicate that the spalling is practically all due to the alpha-beta cristobalite inversion—which takes place at from 220–275° C.[2] In Table 7 is given the data of such a test. From this data it is seen that there is a decided expansion at the lower temperatures. This is followed by a gradual but very slight expansion until a temperature of approximately 1400° C is reached—at which point a rather decided increase in the rate of expansion is noted. This expansion, which becomes apparent at 1400°, is approximately equivalent to the permanent expansion of the brick. Or, in other words, this ex-

[1] *Loc. cit.*

[2] The temperature depends upon the previous heat treatment which the material has received.

TABLE 7.—EXPANSION OF BRICK NO. 27 UNDER A LOAD OF 25 POUNDS PER SQUARE INCH.

Time in hours.	Temperature ° C.	Per cent linear expansion.
0.00	19	0.0
1.00	200	0.0
2.50	500	0.340
3.00	650	0.363
4.50	1200	0.403
4.75	1250	0.408
5.00	1300	0.431
5.25	1330	0.431
5.50	1360	0.657
6.00	1400	0.657
6.50	1400	0.657
7.00	1400	0.703
7.50	1400	0.840
Measured cold after test.....		0.158

Total expansion.....	0.840 Per cent
Permanent expansion.....	0.158 Per cent
Difference compared to 0.657 at 1360° C.....	0.682 Per cent

pansion may be considered as due to the actual transformation of silica from the high to low specific-gravity forms.

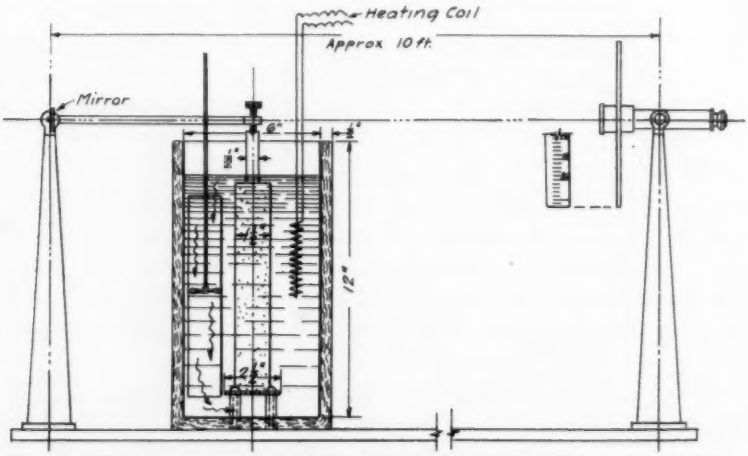

FIG. 6.—Extenseometer, designed to measure the expansion of silica brick between atmospheric temperatures and 300° C.

In Fig. 6 is shown an apparatus for measuring the expansion of silica brick up to 300° C. It consists of a lever arm to register expansion and an attached mirror for magnifying the readings to any desired extent by means of a telescope and graduated scale. The test specimen is immersed in a bath of "Crisco," which is heated electrically by means of a nichrome grid. The temperature throughout the bath is kept uniform by stirring the liquid with a paddle. The supports between the specimen and bed plate are of quartz glass, as is also the rod between the specimen and the lever arm. It would be of interest to determine the exact temperatures at which the chief expansions take place for bricks made from each of the leading varieties of quartzite, and also the time required for a standard 9-inch brick to reach its maximum expansion when the temperature is kept constant.

Load Test.

The load test on silica brick is conducted in the usual form of load test furnace.[1] The load is 25 pounds per square inch. The heating rate is 50° C in 15 minutes from atmospheric temperature to 500° C, 75° C in 15 minutes from 500° C to 800° C, 100° C in 15 minutes from 800° C to 1200° C, 50° C in 15 minutes from 1200° C to 1350° C and 30° C in 15 minutes from 1350° C to 1500° C. A temperature of 1500° C is maintained for $1^1/_2$ hours—after which the firing is discontinued. When cold, the brick should not have increased in length more than two per cent ($^1/_4$″ per foot), nor decreased in length more than one per cent ($^1/_8$″ per foot). The Refractory Materials Committee of the American Gas Institute has set one per cent permanent expansion as the limit in such tests. This one per cent limit is a desirable one if materials which will satisfy it can be regularly obtained.

General Results.

Among the more highly metamorphosed quartzites, those which crush largely by breaking through the original grains are apt to yield brick of lower porosity than those in which the breaking is largely through the interstitial material between the built-up

[1] U. S. Bur. Standards, *Tech. Paper* 7.

grains—or those in which a large proportion of the original grains break free from the interstitial material—while the more friable materials, which crush almost entirely to individual grains, show a very high porosity and yield brick which are weak and friable.

Innovations in the methods of manufacture may be advisable in special cases in order to obtain greater uniformity of product. One such innovation is to control the grind so that the percentages of the various-sized particles in the mix will remain constant at all times and to then machine mold the brick at a pressure of approximately 1500 pounds per square inch in order to obtain brick of smooth finish and uniform size.

The porosity of a finished silica brick depends primarily upon the porosity of the unburned mix. However, in so far as the burning does affect their porosity, the lowest porosity will probably be obtained by not allowing the temperature of the kiln to rise above 1350° C until a large percentage of the quartz has had time to be converted to the lower specific-gravity forms. Rapid heating of a silica brick to temperatures above 1350° C, while a large proportion of the silica is still present as quartz, invariably produces a friable (punky) brick. To obtain a brick of comparatively low specific gravity, however, the temperature should eventually be slowly raised to a maximum corresponding to cones 18–20.

The strength of a brick of slight or medium burn (gauged by present practice) is probably due to the glassy bond formed by the interaction of the basic fluxes with the silica, while in well burned brick it appears that the strength is greatly augmented by the formation of interlocking crystals of the low specific-gravity forms of silica. As a general rule, it has been found that silica brick, made from quartzite giving entire satisfaction in other ways, usually meet the strength requirements of service.

Inspection of Ware Based on the Above Observations.

Some of the properties of a finished brick which may be used in judging its probable value in use are as follows: Chemical composition, specific gravity, porosity, cold cross-breaking strength, behavior in the load test, the hot crushing strength

(at the temperature at which bricks are to be used), and the amount of interlocking crystals—determined by viewing thin sections of the brick under the microscope—and the softening temperature of the brick. For the best quality brick these properties, based on available data, approximate the following:

The silica content should not be much under 94 per cent. Bricks containing lower percentages of silica are less refractory. The alkalies should not exceed 0.5 per cent. Greater percentages reduce the refractoriness in direct proportion to their amount. The limit of iron oxide was previously assumed to be 0.5 per cent. However, greater percentages, up to 1.6 per cent, do not appear to materially affect the refractoriness of the brick. The limit for lime is usually set at 2.0 per cent, although larger amounts of lime (up to several per cent) do not greatly lower the softening temperatures. However, in commercial practice, more than 2.0 per cent of lime is considered undesirable.

The average specific gravity for brick made from Medina quartzite, as shown in Table 3, is 2.357 and, for brick made from Baraboo quartzite, it is 2.399. Hence, bricks made from similar materials may be considered as having received a medium burn if their specific gravities correspond to these figures.

The practical limits of porosity which first quality silica brick are apt to have are, Medina 22.80 to 31.52 per cent, and Baraboo 23.45 to 25.66 per cent. However, porosities lower than these might be desirable.

The cold cross-breaking tests, as shown in Table 4 and from other data, should not show an average modulus of rupture much below 500 pounds per square inch. The reliability of this figure is considerably impaired by the comparatively small amount of data upon which it is based.

After having been tested under a load of 25 pounds per square inch at 1500° C, a brick should have neither expanded (linearly) more than two per cent (preferably 1.0 per cent), nor contracted more than one per cent. If desired, the load test furnace may be arranged so that the specimen can be crushed hot at the completion of the test. It would seem desirable to conduct this test at the temperature at which the bricks are to be used.

A micro-examination of a thin section of a brick will detect the amounts of interlocking crystals of the low specific-gravity forms which are present, and thus indicate the degree of burning which the brick received in manufacture.

First quality brick should not have a fusion temperature much below that of cone 31.[1] Cones of the most refractory silica brick bend over at cone $32\frac{1}{2}$.

Summary.

In summing up, we may say that the bulk of all silica bricks are made from the quartzites of early geologic age, and that, as a rule, the quartzites most suitable for the manufacture of silica brick are those which range from the slightly porous to the highly metamorphosed impervious rocks—having tightly interlocking grains. These may be distinguished in thin section under the microscope, by the fact that the interstitial impurities (such as hydrated iron oxide) appear as sharply defined lines between the tightly interlocking quartz grains, while in material which has been but slightly metamorphosed the interstitial material appears in translucent, cloudy areas between the harder quartz grains. Those quartzites which crush largely by a breaking through of the original grains are apt to yield silica brick of lower porosity than those in which the breaking is largely through the interstitial material between the built up grains, or those in which the original grains break free from the interstitial material.

In special cases, to obtain greater uniformity of product, it may be advisable to control the grind by unusual manipulation—so that the percentages of various-sized particles in the mix will remain constant at all times—and to then mold the brick by machine.

The porosity of a finished silica brick depends primarily upon the porosity of the unburned mix. However, the heating of brick, which contain large percentages of unchanged quartz, to temperatures above 1350° C causes undue expansion—resulting in a punky product. On the other hand, maintaining the tempera-

[1] When ground to pass through a 60-mesh screen and made into cones similar to the standard cones used for comparison.

ture of the kiln at from 1250° C to 1350° C, until a large percentage of the quartz in the bricks has been transformed to the low specific-gravity forms, and then slowly proceeding to the higher temperatures, should result in a low porosity brick of low specific gravity.

The specific gravity of a silica brick, as determined by the wet, dry and suspended weight method, answers as a quick means of determining the degree to which the quartz originally present has been transformed to the lower specific-gravity forms of silica. But to obtain a comprehensive idea of the qualities of any variety of silica bricks, the other properties, such as porosity, cold cross-breaking strength, softening temperature, behavior in the load test, and appearance in thin section under the microscope should also be determined.

COMMUNICATED DISCUSSIONS.

R. M. HOWE: Mr. Ross mentions a great many interesting points arising in the field of silica brick manufacture and gives considerable valuable information.

An interesting point is in connection with his screen analyses as applied to "sand rock." A typical sand-rock analysis follows:

	Per cent.
Retained on 14-mesh sieve	0.73
" " 20 " "	2.02
" " 40 " "	42.55
" " 60 " "	46.30
" " 80 " "	7.12
" " 100 " "	0.92
" " 150 " "	0.39
" " 230 " "	0.21
Through 230-mesh sieve	0.20
Total	100.44

He also mentions the rapidly growing importance of the "specific-gravity" test. Its value is recognized at the present time to such an extent that one company finishes all kilns by means of it. The draw trials are taken from the kilns at intervals and when they have a specific gravity of 2.40 the firing is stopped.

As the kiln is held the brick undergo further transformation—the specific gravities of the brick when drawn being below 2.38. Records are carefully kept, and the firing of each particular kiln may be controlled by this means after sufficient data has been collected.

Mr. Ross, however, makes some statements regarding chemical analysis which are evidently not justified by the work completed to the present time. There is no evidence on record which proves that certain impurities cannot be tolerated. Some materials are high in one particular impurity, but are correspondingly low in other impurities. In view of this and of the necessary incriminating evidence, one cannot justly say as to what percentage of each individual impurity may or may not be tolerated.

As a rule, however, the total percentages of impurities in good grades of silica brick are similar. These generally run between 4.0 and 6.5 per cent. If, then, the total of silica averages in the vicinity of 94.0 per cent or more, about all has been done which can be done. A silica content of 94.0 per cent excludes any superabundance of impurity and yet does not unjustly discriminate against any particular combination of the same. It appears that suitable material can be secured on a silica basis alone. In so doing, the usual exceptions encountered when specifying ceramic materials on the analytical basis will be avoided.

The tests recommended by Mr. Ross have the same general aim. They all tend to develop a well-made, well-burned brick. It is surprising to note how well these same features can be determined instantly by one familiar with a particular brand of silica brick. Two bricks when tapped together show by their ring nearly as much as can be learned by tests. This may be explained by the fact that the raw material is practically the same—the lime bond being kept within narrow limits and most silica brick being sufficiently refractory. The "ring" test determines the variable qualities—the burn and structure. The specific-gravity test, however, goes beyond the scope of the "ring" test in that it is accurate in discriminating between different brands. Some raw materials burn to form a product of good "ring" at low

temperatures. Such materials are quickly detected because of their high specific gravities. This is found to be particularly true when checked by the re-heating (expansion) test.

D. W. Ross: With reference to Mr. Howe's remarks on the above paper, it is of interest to note that in his screen analysis, in determining the size of grain in sandrock (presumably either Homewood sandstone or Oriskany sandstone) large percentages were retained on the 40- and 60-mesh sieves. This apparently checks the results of our own screen analyses.

It is gratifying to know that in so short a time the specific-gravity test for silica brick has been applied in so many useful ways, not the least of which is the method for control of the burning as set forth by Mr. Howe.

Mr. Howe strongly emphasizes the point brought out in the paper, that silica brick, as manufactured in the United States, usually fail on account of their physical properties and not on account of their chemical composition. In our opinion, entirely too much importance has, in the past, been placed on the chemical composition. With this in mind, no presentation of data and detailed discussion has been given in reference to chemical composition. Instead, the merest statement of the results obtained by ourselves and others, and of the usual practice as we understand it, have been set forth. This has been done in order to place on record information which we hope may be of assistance to future investigators in this complex and interesting field—the effects of fluxes, etc., on the properties of silica brick.

ANTIMONY OXIDE AS AN OPACIFIER IN CAST IRON ENAMELS.

By J. B. Shaw, Alfred, N. Y.

Introduction.

The use of antimony oxide in the replacement of tin oxide as an opacifying agent in cast iron enamels has been the subject of a number of investigations. The subject has always been an important one, but there has never been a time when it was so vital as at the present. The present market price of tin oxide makes its use in enamels almost prohibitive. Eight per cent is about the minimum amount of tin oxide that will give satisfactory results in cast iron enamels. At 72 cents per pound, this means that the tin oxide alone in 100 lbs. of raw enamel will cost $5.76. This was the cost of 100 lbs. of enamel used by the writer in 1913.

Antimony compounds (Leukonin) may be purchased for 16 cents per lb. and antimony oxide is relatively cheap. When it is considered that antimony oxide is equal to tin oxide, pound for pound, as an opacifying agent, it is readily seen that the manufacturers of cast iron enamel wares have a possibility of reducing the cost of their enamels about 100 per cent by the use of antimony oxide.

Staley[1] has presented a very clear statement of the difficulties and possibilities of the problem together with the results of the experiments he has performed. It is not the intention to enter into a detailed discussion of the problem or to repeat what can be readily obtained from the above-mentioned article. Much of the data given herein confirms Staley's conclusions—although on some points our opinions are contradictory. This article will have served its purpose if it stimulates some manufacturer to experiments which result in the successful use of antimony compounds in his enamels.

Antimony oxide has been used successfully as an opacifier in

[1] *Trans. Am. Ceram. Soc.*, **18**, 173 (1915).

steel enamels for many years. There is no material difficulty attending its use in the frit for steel enamels, the color being a minor consideration as compared with cast iron enamels—the other constituents of the steel enamel being such as to give better color than those of the cast iron enamels. It may be said that there are no great difficulties attending the use of antimony oxide in cast iron enamels, aside from the obtaining of satisfactory colors. The color, however, is of the first importance—so great in fact that even now, after years of experimenting and when the desirability for its use is so great, antimony oxide is used only in very limited amounts. However, its use is bound to be extended with an increase in the knowledge of how to modify formulas so as to secure satisfactory results.

The results described were obtained in an effort to outline satisfactory working formulas—having antimony oxide as the chief opacifying agent—for commercial use. All have reference to cast iron enamels.

Experimental.

The work was carried out in three stages as follows:

(1) Preliminary batches, hundreds of which were made, were melted in 500-gram quantities in a laboratory furnace. Denver fire clay crucibles were found quite satisfactory for this purpose. It was generally necessary to insert the cold crucible with its batch into the furnace at a temperature of from 1100° to 1300° C. During all of the experiments it was very seldom that the fire-clay crucibles failed under the treatment.

(2) After making laboratory trials with the enamels, those which gave promise of satisfactory results were melted in batches of about 75 lbs. The melted batches were ground and applied on commercial wares. The preliminary melts gave a good indication as to the results which were likely to be obtained with full-sized batches—a great amount of time, money and material being thereby saved.

(3) The enamels were finally melted in batches of about 2000 pounds and applied to commercial ware.

The materials used in compounding the enamels were all of a good commercial grade, the formulas given being figured to a basis of chemically pure materials (feldspar included). A Connecticut feldspar having the following analysis was used:

Chemical analysis.	Per cent.
K_2O	7.30
Na_2O	2.90
Al_2O_3	18.60
SiO_2	70.40
Fe_2O_3	0.14
CaO	0.65
MgO	trace
Loss	0.26
	100.25

Formula

$$\left.\begin{matrix} 0.62\ K_2O \\ 0.38\ Na_2O \end{matrix}\right\} 1.4\ Al_2O_3 \left\{ 9.4\ SiO_2 \right.$$

Formula weight, 790.0.

Series 1.

Knowing the efficiency of cryolite as a flux and opacifier in enamels, a number of trials were made with a view to determining the possibility of using it in conjunction with antimony oxide. To this end the following series of enamels was prepared:

SERIES 1.—BATCH WEIGHTS.

No.	Feld-spar.	Flint.	Borax.	Soda ash.	Niter.	Fluor-spar.	Cryo-lite.	Barium carbon-ate.	Zinc oxide.	Anti-mony oxide.	PbO.
1	53	0	58	21	26	8	42	0.0	0.0	2.5	0.0
2	73	0	58	28	26	8	29	0.0	0.0	2.5	0.0
3	84	0	58	32	26	8	21	0.0	0.0	2.5	0.0
4	56	24	58	21	25	8	42	0.0	0.0	2.5	0.0
5	73	24	58	28	26	8	29	0.0	0.0	2.5	0.0
6	84	24	58	32	26	8	21	0.0	0.0	2.5	0.0
7	56	54	58	21	26	8	42	0.0	0.0	2.5	0.0
8	73	54	58	28	26	8	29	0.0	0.0	2.5	0.0
9	84	54	58	32	26	8	21	0.0	0.0	2.5	0.0
10	56	0	58	11	26	16	42	0.0	0.0	2.5	0.0
11	73	0	58	17	26	16	29	0.0	0.0	2.5	0.0
12	84	0	58	21	26	16	21	0.0	0.0	2.5	0.0
13	56	24	58	11	26	16	42	0.0	0.0	2.5	0.0
14	73	24	58	17	26	16	29	0.0	0.0	2.5	0.0
15	84	24	58	21	26	16	21	0.0	0.0	2.5	0.0
16	56	54	58	11	26	16	42	0.0	0.0	2.5	0.0
17	73	54	58	17	26	16	29	0.0	0.0	2.5	0.0

SERIES 1.—BATCH WEIGHTS—(*Continued*).

No.	Feld-spar.	Flint.	Borax.	Soda ash.	Niter.	Fluor-spar.	Cryo-lite.	Barium carbon-ate.	Zinc oxide.	Anti-mony oxide.	PbO.
18	84	54	58	21	26	16	21	0.0	0.0	2.5	0.0
19	56	0	58	0	26	23	42	0.0	0.0	2.5	0.0
20	73	0	58	6	26	23	29	0.0	0.0	2.5	0.0
21	84	0	58	11	26	23	21	0.0	0.0	2.5	0.0
22	56	24	58	0	26	23	42	0.0	0.0	2.5	0.0
23	73	24	58	6	26	23	29	0.0	0.0	2.5	0.0
24	84	24	58	11	26	23	21	0.0	0.0	2.5	0.0
25	56	54	58	0	26	23	42	0.0	0.0	2.5	0.0
26	73	54	58	6	26	23	29	0.0	0.0	2.5	0.0
27	84	54	58	11	26	23	21	0.0	0.0	2.5	0.0
28	110	0	58	0	24	23	29	0.0	0.0	3.0	0.0
29	73	25	76	5	19	23	29	0.0	0.0	3.0	0.0
30	73	43	95	0	19	23	29	0.0	0.0	3.0	0.0
31	73	43	58	11	19	16	29	20.0	0.0	3.0	0.0
32	73	43	58	11	19	16	29	0.0	8.0	3.0	0.0
33	73	43	58	0	19	16	29	20.0	8.0	3.0	0.0
34	73	43	67	8	2	12	29	20.0	12.0	5.0	0.0
35	73	43	76	6	2	12	29	20.0	12.0	5.0	0.0
36	73	43	86	3	2	12	29	20.0	12.0	5.0	0.0
37	73	43	95	0	2	12	29	20.0	12.0	5.0	0.0
41	56	66	57	0	7	8	29	20.0	16.0	5.0	22.0
42	56	66	57	0	7	8	29	20.0	20.0	5.0	11.0
43	56	66	57	0	7	8	29	0.0	28.0	5.0	11.0
44	90	0	57	15	17	8	0	0.0	24.0	8.0	11.0

SERIES 1.—FORMULAS.

No.	KNaO.	CaO.	BaO.	ZnO.	Al_2O_3.	SiO_2.	B_2O_3.	PbO.
1	0.9	0.10	0.0	0.0	0.20	0.60	0.30	0.0
2	0.9	0.10	0.0	0.0	0.20	0.60	0.30	0.0
3	0.9	0.10	0.0	0.0	0.20	0.60	0.30	0.0
4	0.9	0.10	0.0	0.0	0.20	1.00	0.30	0.0
5	0.9	0.10	0.0	0.0	0.20	1.00	0.30	0.0
6	0.9	0.10	0.0	0.0	0.20	1.00	0.30	0.0
7	0.9	0.10	0.0	0.0	0.20	1.50	0.30	0.0
8	0.9	0.10	0.0	0.0	0.20	1.50	0.30	0.0
9	0.9	0.10	0.0	0.0	0.20	1.50	0.30	0.0
10	0.8	0.20	0.0	0.0	0.20	0.60	0.30	0.0
11	0.8	0.20	0.0	0.0	0.20	0.60	0.30	0.0
12	0.8	0.20	0.0	0.0	0.20	0.60	0.30	0.0
13	0.8	0.20	0.0	0.0	0.20	1.00	0.30	0.0
14	0.8	0.20	0.0	0.0	0.20	1.00	0.30	0.0

SERIES 1.—FORMULAS—(*Continued*).

15	0.8	0.20	0.0	0.0	0.20	1.00	0.30	0.0
16	0.8	0.20	0.0	0.0	0.20	1.50	0.30	0.0
17	0.8	0.20	0.0	0.0	0.20	1.50	0.30	0.0
18	0.8	0.20	0.0	0.0	0.20	1.50	0.30	0.0
19	0.7	0.30	0.0	0.0	0.20	0.60	0.30	0.0
20	0.7	0.30	0.0	0.0	0.20	0.60	0.30	0.0
21	0.7	0.30	0.0	0.0	0.20	0.60	0.30	0.0
22	0.7	0.30	0.0	0.0	0.20	1.00	0.30	0.0
23	0.7	0.30	0.0	0.0	0.20	1.00	0.30	0.0
24	0.7	0.30	0.0	0.0	0.20	1.00	0.30	0.0
25	0.7	0.30	0.0	0.0	0.20	1.50	0.30	0.0
26	0.7	0.30	0.0	0.0	0.20	1.50	0.30	0.0
27	0.7	0.30	0.0	0.0	0.20	1.50	0.30	0.0
28	0.7	0.30	0.0	0.0	0.20	1.20	0.30	0.0
29	0.7	0.30	0.0	0.0	0.27	1.20	0.40	0.0
30	0.7	0.30	0.0	0.0	0.20	1.50	0.50	0.0
31	0.7	0.20	0.1	0.0	0.20	1.50	0.30	0.0
32	0.7	0.20	0.0	0.10	0.20	1.50	0.30	0.0
33	0.6	0.20	0.1	0.10	0.20	1.50	0.30	0.0
34	0.6	0.15	0.1	0.15	0.20	1.50	0.35	0.0
35	0.6	0.15	0.1	0.15	0.20	1.50	0.40	0.0
36	0.6	0.15	0.1	0.15	0.20	1.50	0.45	0.0
37	0.6	0.15	0.1	0.15	0.20	1.50	0.50	0.0
41	0.1	0.40	0.1	0.20	0.17	1.70	0.30	0.10
42	0.1	0.40	0.1	0.25	0.17	1.70	0.30	0.05
43	0.1	0.40	0.0	0.35	0.17	1.70	0.30	0.05
44	0.1	0.39	0.0	0.30	0.16	0.96	0.30	0.05

Results—Series 1.

Enamels Nos. 1 to 27 were first tested on small trials. All of these are very fusible with the exception of Nos. 25, 26 and 27. The most brilliant enamels were those highest in silica and lowest in cryolite. Very dull (matt) enamels resulted from high cryolite and low silica contents. The color was fairly good throughout the series but those having the highest gloss—highest silica—generally had the best white color.

The solubility of all of the enamels was determined by partly immersing in water and subjecting them to 15 pounds' steam pressure for 2 hours in an autoclave. The results were as follows:

Nos. 1 to 6—Very badly decomposed.
Nos. 7 to 8—Much less than preceding.
Nos. 7 to 9—Hardly noticeably affected.
Nos. 10 to 12—Badly etched.
Nos. 13 to 15—Very slightly affected.
No. 16—Distinctly etched.
Nos. 17 to 27—Unaffected.

The above results may be interpreted as follows:

A. To increase the fusibility: (1) increase alkalies, (2) increase cryolite, (3) decrease silica.
B. To increase lustre: (1) decrease cryolite, (2) increase silica.
C. To increase opacity: increase the content of cryolite and antimony.
D. To decrease solubility: (1) increase fluorspar,[1] (2) decrease cryolite, (3) increase silica.
E. To purify color: (1) decrease cryolite, (2) increase silica.

After studying the enamels of the series up to No. 27, 75-pound batches of Nos. 23 and 28 were made and applied on commercial ware. The color and opacity were good but both crazed very badly—showing the influence of cryolite in producing crazing. The silica content of these enamels is much higher than that of good commercial enamels not containing cryolite.

Enamels Nos. 29 to 33, inclusive, respresent the attempts made to cure the crazing in No. 28. These are all fine white enamels having a high luster. When tested for solubility, No. 32 showed no mark and No. 33 was only slightly dulled in a 2-hour autoclave test.

Nos. 29, 30 and 31 are somewhat decomposed but decidedly superior to Nos. 23 and 28. Nos. 32 and 33 were both too hard and lift or craze in the curves when applied on commercial ware. Nos. 34 to 37 were made in an attempt to cure crazing by an increase of the boric acid content. The crazing gradually decreased up to No. 36, which fitted the iron but was decidedly lacking in covering power.

It was therefore decided that in order to retain the cheapness and stability of the enamel, crazing must be corrected by increasing the silica content. This was attempted, as is represented by enamels Nos. 41 to 43, which crazed and were decidedly harder (more infusible) than the ordinary cast iron enamels. In

[1] This holds true only when smelting is very complete.

order to get a sufficient thickness of enamel to give a good white coat, three or more dredgings were necessary. This required a departure from the usual factory practice and the results were not sufficiently promising to justify their adoption.

Series 2.

The results obtained with the cryolite enamels led to the conclusion that, while good colors were easily obtained, it would not be possible to utilize the fluxing power of cryolite to advantage and that the range of composition within which they would fit the iron would be small. In other words, the factor of safety in cryolite enamels is low. Because of this fact it was deemed advisable to dispense with the use of cryolite and develop satisfactory formulas without it.

To this end the following series was prepared:

SERIES 2.—BATCH WEIGHTS.

No.	Feldspar.	Borax.	Soda ash.	Niter.	Barium carbonate.	Zinc oxide.	Antimony oxide.	Litharge.
47	78	57	49	17	0	8	20	11
48	78	57	39	17	0	16	20	11
49	78	57	29	17	0	20	20	11
50	78	57	49	17	20	0	20	11
51	78	57	39	17	40	0	20	11
52	78	57	29	17	60	0	20	11
53	78	57	49	17	10	4	20	11
54	78	57	39	17	20	8	20	11
55	78	57	29	17	30	12	20	11
56	78	57	22	17	30	16	20	11
57	78	57	17	17	30	20	20	11

FORMULAS.

No.	K_2O.	Na_2O.	BaO.	ZnO.	Al_2O_3.	SiO_2.	B_2O_3.	Sb_2O_3.	PbO.
47	0.14	0.71	0.0	0.10	0.14	0.84	0.3	0.07	0.05
48	0.14	0.61	0.0	0.20	0.14	0.84	0.3	0.07	0.05
49	0.14	0.51	0.0	0.30	0.14	0.84	0.3	0.07	0.05
50	0.14	0.71	0.10	0.0	0.14	0.84	0.3	0.07	0.05
51	0.14	0.61	0.20	0.0	0.14	0.84	0.3	0.07	0.05
52	0.14	0.51	0.30	0.0	0.14	0.84	0.3	0.07	0.05
53	0.14	0.71	0.05	0.05	0.14	0.84	0.3	0.07	0.05
54	0.14	0.61	0.10	0.10	0.14	0.84	0.3	0.07	0.05
55	0.14	0.51	0.15	0.15	0.14	0.84	0.3	0.07	0.05
56	0.14	0.46	0.15	0.20	0.14	0.84	0.3	0.07	0.05
57	0.14	0.41	0.15	0.25	0.14	0.84	0.3	0.07	0.05

Results—Series 2.

This series (Nos. 47 to 57) was a total failure. It is given here in order to bring out the one point of interest which it revealed. All preceding and subsequent enamels in this investigation were first melted in small crucible batches. These 10 enamels were prepared as usual and placed in the furnace, but not one of them could be melted down to a homogeneous solid enamel. All frothed and boiled vigorously and retained the consistency of soap suds after intense heat treatment. A repetition of the series gave the same results. A satisfactory explanation of this phenomenon has not been found. It will be noted that the enamels contained no lime and it was found that a very slight addition of either fluorspar or whiting would cause the enamels to fuse down normally. It has been noted in many cases, since this observation was made, that enamels containing no lime or cryolite are likely to be frothy and smelt with great difficulty and are also likely to repeat this boiling when burned on the ware.

Observations made on this point in many instances substantiate the statement that no very satisfactory enamel can be made in the absence of lime (fluorspar or calcium carbonate), lead or cryolite.

Series 3.

The enamels of the following series represent the type of enamel which has been found by the writer to give the best results when antimony oxide is used as the opacifier:

SERIES 3.—BATCH WEIGHTS.

No.	Feldspar.	Borax.	Soda ash.	Niter.	Fluorspar.	Litharge.	Barium carbonate.	Zinc oxide.	Antimony oxide.
58	78	57	39	17	8	11	0	8	17
59	78	57	29	17	8	11	0	16	17
60	78	57	19	17	8	11	20	16	17
61	78	57	6	17	8	11	40	16	17
62	90	57	15	17	8	11	0	24	17
63	95	57	15	17	8	11	0	24	17
64	100	57	13	17	8	11	0	24	17
65	84	57	17	17	8	0	10	24	17
66	90	57	15	17	8	0	10	24	17

	Formulas.								
No.	K_2O.	Na_2O.	CaO.	BaO.	ZnO.	PbO.	Al_2O_3.	SiO_2.	B_2O_3.
58	0.14	0.61	0.10	0.0	0.10	0.05	0.14	0.84	0.3
59	0.14	0.51	0.10	0.0	0.20	0.05	0.14	0.84	0.3
60	0.14	0.41	0.10	0.10	0.20	0.05	0.14	0.84	0.3
61	0.14	0.31	0.10	0.20	0.20	0.05	0.14	0.84	0.3
62	0.16	0.39	0.10	0.0	0.30	0.05	0.16	0.96	0.3
63	0.17	0.38	0.10	0.0	0.30	0.05	0.17	1.02	0.3
64	0.18	0.37	0.10	0.05	0.30	0.05	0.18	1.08	0.3
65	0.15	0.40	0.10	0.05	0.30	0.0	0.15	0.90	0 3
66	0.16	0.39	0.10	0.10	0.30	0.0	0.16	0.96	0.3

The control of crazing and shivering is best accomplished by varying the silica content. The fusibility of these enamels is quite similar to that of the ordinary tin enamels and the opacity is quite as good as that of enamels in which an equal amount of tin oxide is used.

The color depends largely on the purity and color of the antimony oxide used. Unless the very purest white oxide is used, the color of the enamels is liable to be similar to that of the oxide itself.

Excessive smelting will produce a bluish green color in any antimony enamel containing fluorspar and a low percentage of lead.

A high lead content is not permissible with antimony because the combination produces a yellow color. Blue-white can be converted to pinkish or yellow-white by the careful addition of Venetian Red or Prince's Metallic Brown (ferric oxide) up to 0.4 per cent of the weight of the raw batch.

Thoroughly oxidizing conditions (plenty of niter) must be maintained at all times during the smelting of antimony enamels. Reduction causes the development of a green color. Manganese dioxide (up to 0.4 per cent) may be used to mask the blue-green color of the fluorspar-antimony compound. An ivory-green color results from the use of low fluorspar or insufficient smelting or both. The addition of 1.0 per cent of lead oxide will sometimes convert a useless leadless antimony enamel to one having a pleasing cream color.

Conclusion.

In order to produce antimony enamels of satisfactory color the following should be emphasized:

1. Use only the purest, white, antimony oxide.
2. Use a little fluorspar (not over 5 per cent of the raw batch).
3. Be sure to maintain oxidizing conditions during smelting (use plenty of niter).
4. Control the color by the addition of ferric oxide, manganese dioxide and lead oxide.
5. Cryolite is unsatisfactory as a flux or opacifier in antimony enamels but small quantities (up to 3 per cent) will aid in producing a good color and without introducing any troublesome element.
6. Extreme care in proportioning the raw materials, very careful and thorough mixing, and proper smelting, will insure the successful use of antimony oxide.

ALFRED UNIVERSITY,
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DISCUSSION.

E. P. POSTE: Mr. Shaw's paper brings to mind the work of Staley on the control of crazing in cast iron enamels, in which he mentioned the theoretical consideration of the coefficient of expansion. I wonder if Mr. Shaw, or anyone else, has had experience in a case such as he mentioned, *i. e.*, of merely increasing the silica content in order to slightly modify the entire formula so that the theoretical coefficient of expansion will remain the same and in such a way as to retain the same fusibility and at the same time retain the same theoretical coefficient of expansion.

MR. STALEY: I did not understand the remark that was made with reference to my paper.

MR. SHAW: I made the statement that cryolite is not detrimental to the color of antimony enamels; that fluorine, introduced as cryolite, does not produce an unsatisfactory color, and that this is in contradiction to the opinions expressed by Mr. Staley and published in a preceding volume of the Transactions.

MR. STALEY: There seems to be some misunderstanding of my statement. I stated that the objectionable blue color of some antimony oxide enamels is due to a combination of fluorine, boric oxide, calcium and antimony, and that if the calcium and

antimony are in certain proportions and the percentage of fluorine increased—whether by the addition of fluorspar or cryolite—an objectionable blue color will result. I have used large quantities of cryolite in oxide of antimony enamels—in fact to my knowledge practically all of the recipes which are being used commercially for making oxide of antimony enamels contain cryolite.

As near as I can remember, in the empirical formulas of the oxide of antimony enamels, which I know have been used in factory practice, the total amount of sodium and potassium ordinarily found in American enamels falls, in nearly every case, between 0.4 and 0.5 equivalent. The silica content of antimony oxide enamels in this country varies from about 0.75 up to possibly 1.1 equivalents. Mr. Shaw has developed a type of enamel higher in alkalies than those commonly used in this country.

MR. SHAW: I would like to ask Mr. Staley: how would he secure opacity from cryolite in an enamel containing 0.3 equivalent of KNaO? if he had enough cryolite to secure any fluxing action, how would he keep the KNaO down to 0.3 equivalent?

MR. STALEY: When the total is as low as 0.3 equivalent, the alkali practically all comes from the feldspar and the fluorine is introduced as fluorspar, but if the total alkalies run up to 0.4 equivalent, practically 0.1 equivalent may come from cryolite. The cryolite in percentage weight will run around about 5 per cent of the melted weight of the enamel. Large percentages are not commonly used.

MR. SHAW: I recommend in the formulas that some cryolite, 1 to 3 per cent, be used. You will not secure opacity with 5 per cent of cryolite. If you use less than 8 to 10 per cent of the raw weight, you do not get much fluxing or opacifying value. Referring to the last slide on the screen. Here is shown the type of formula which I not only have been using but am using today on cast iron enamels. I am not stating that anyone here can take any one of these formulas and make a cast iron enamel; that is, at the first trial. There are a number here who can work out a satisfactory enamel from any one of the formulas shown, but

they will have to correct for color, as I stated in the paper. The color is the troublesome part. Here are shown formulas of enamels containing from 0.45 equivalent KNaO up to 0.75 equivalent KNaO. I can take any formula shown and make a satisfactory cast iron enamel. There is no cryolite in any of them, but 3 per cent of cryolite may be added without any detriment to the enamel. I am unable to see the benefits to be derived from the use of cryolite.

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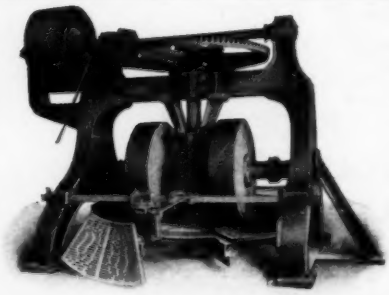

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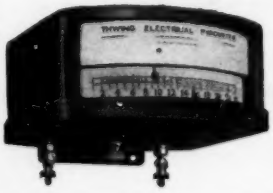

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